THE JOURNAL
OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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CONTENTS:

Bureau of Employment - - - - - - - - - 144

Austrian Leather Technicians Society - - - - - 144

Council Meeting Minutes - - - - - - - - 144

Report from the Technical Committees of the A.L.C.A. - 150

Report of the Committee on Joint Action with the ASTM 151

Tanning Studies with Epoxy Resins *By* E. M. FILACHIONE and E. H. HARRIS, JR. - - - - - - - - 160

The Chemistry of Vegetable Tannins. XIV Wattle. - *By* R. C. PUTNAM - - - - - - - - - 169

Studies on Leather by Means of Sonic Technique *By* J. R. KANAGY and M. ROBINSON - - - 174

Abstracts - - - - - - - - - - - - 198

Patents - - - - - - - - - - - - 200

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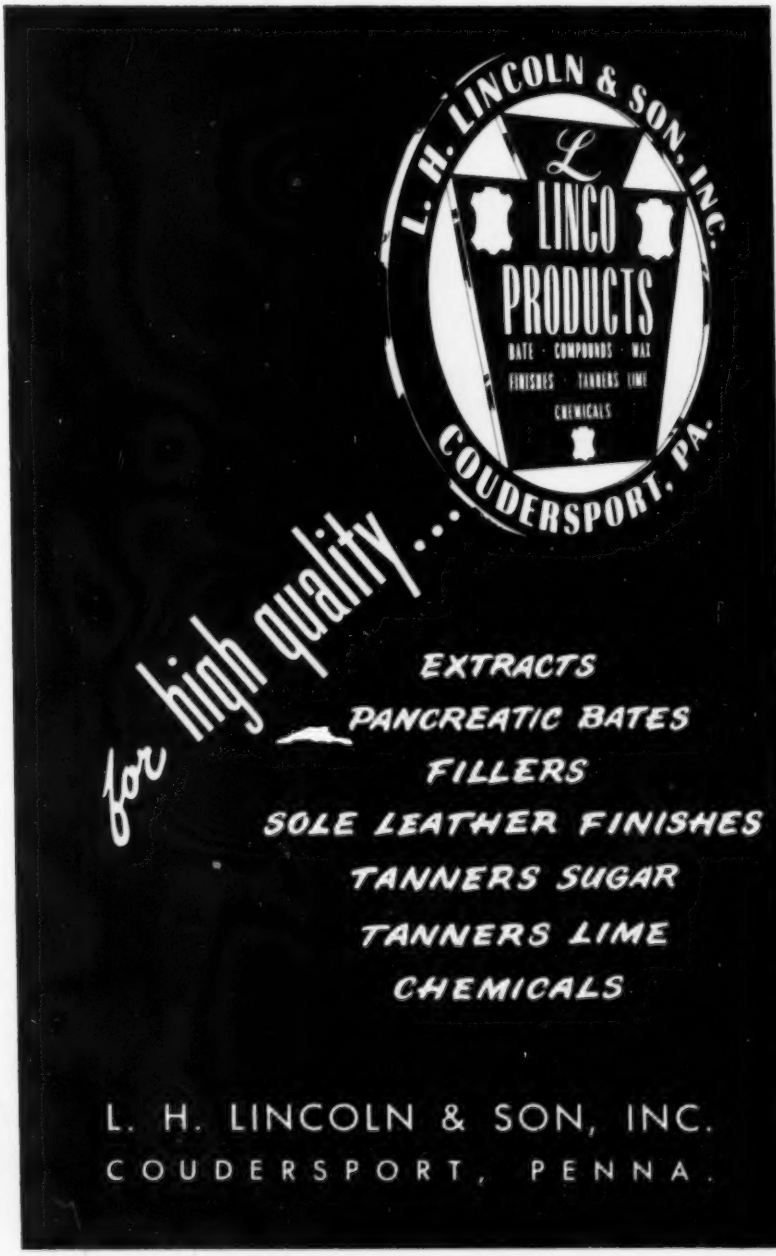
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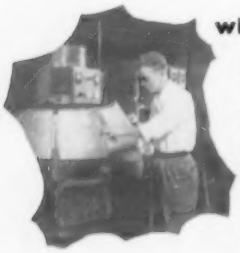

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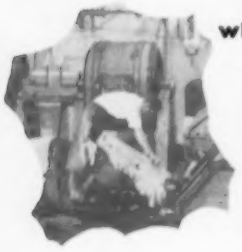

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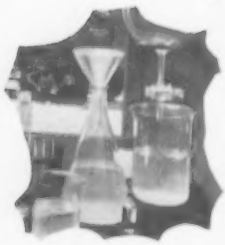

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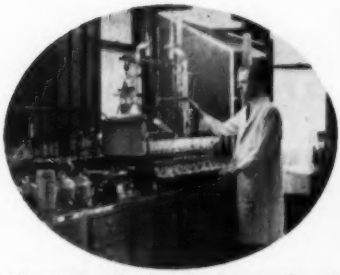

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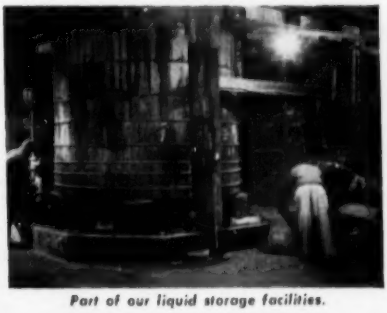

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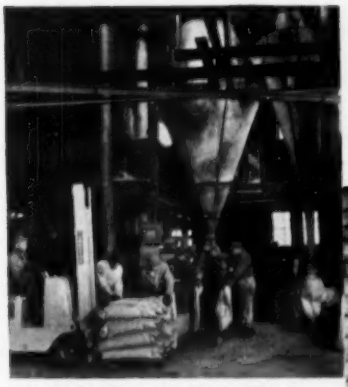

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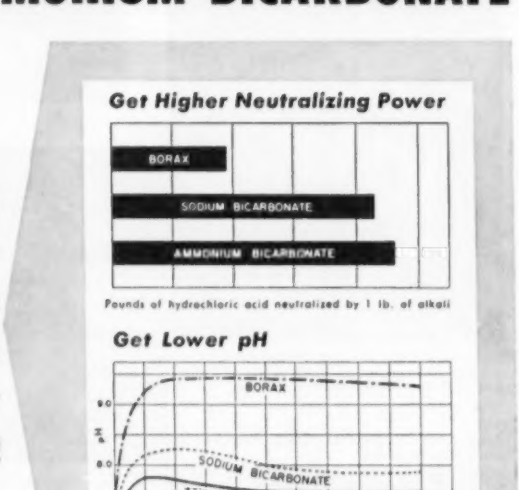

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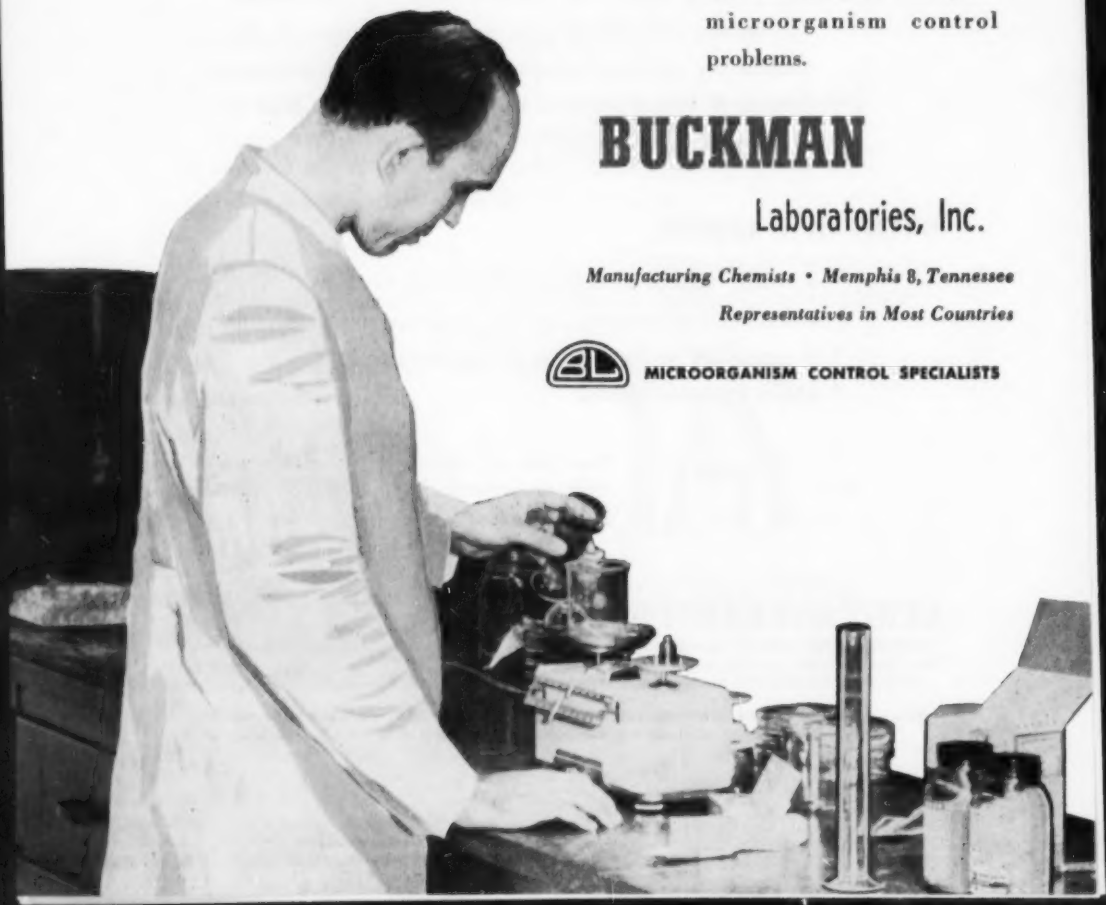

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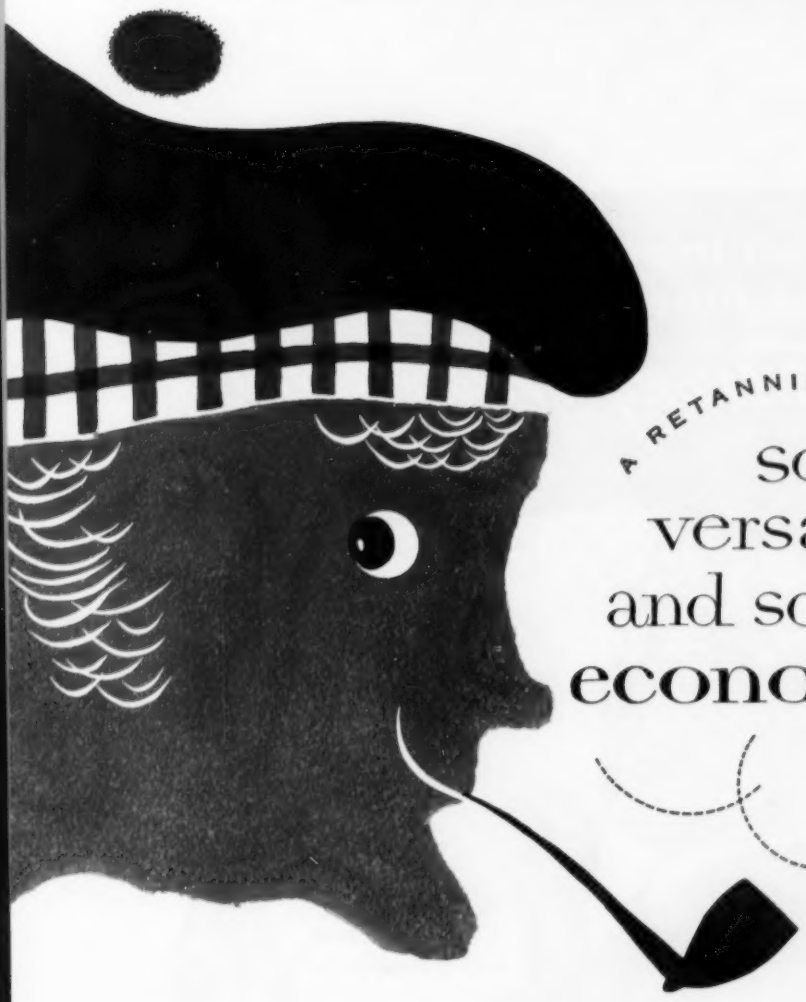

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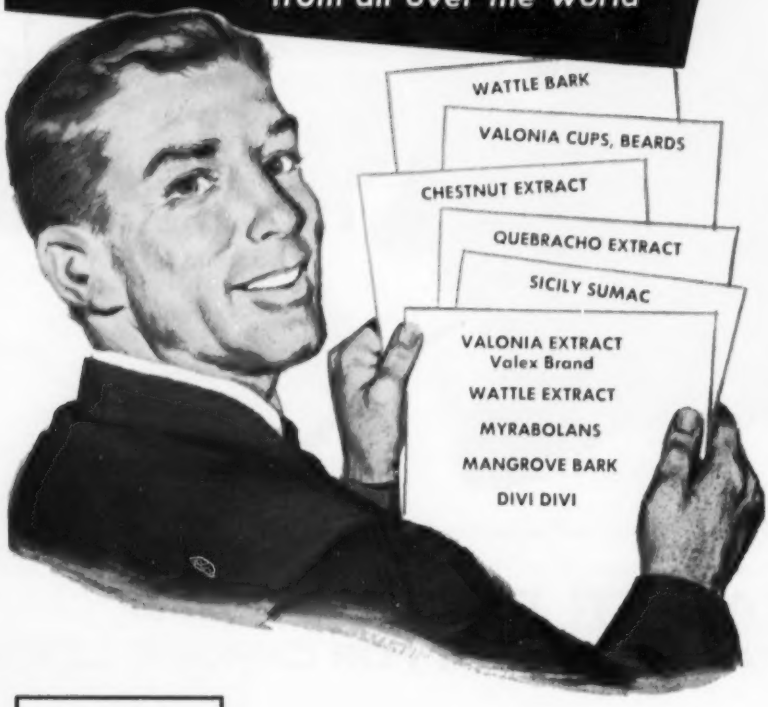

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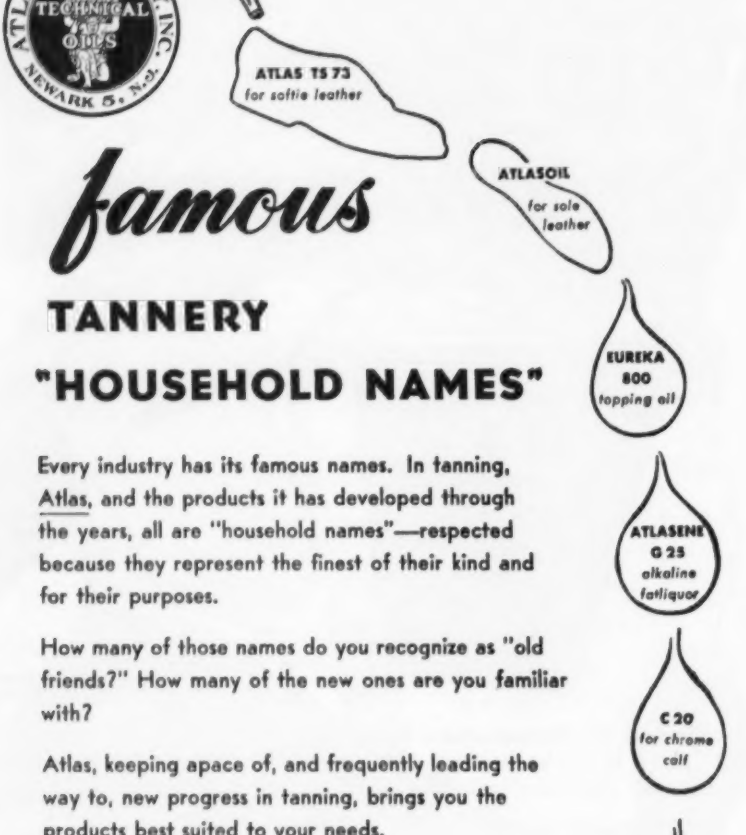

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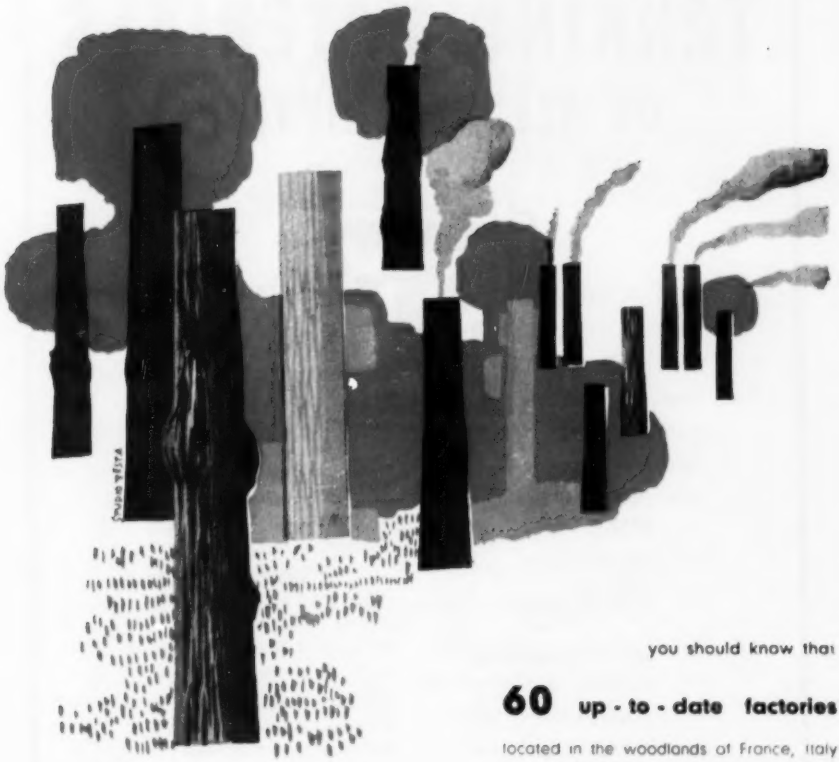

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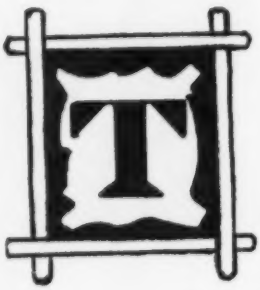

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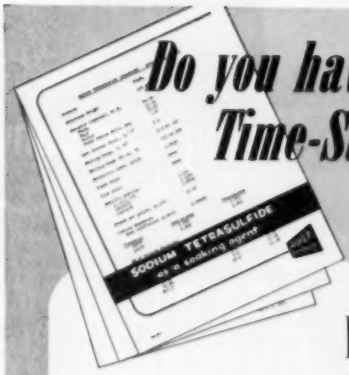

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JOURNAL OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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AUSTRIAN LEATHER TECHNICIANS SOCIETY

The Austrian Leather Technicians Society will hold their meeting in Vienna, Austria on May 2 to May 5. Our members are invited to participate and they may offer to present papers at this meeting.

COUNCIL MEETING MINUTES

AMERICAN LEATHER CHEMISTS ASSOCIATION

January 13, 1956

Hotel Plankinton

Milwaukee, Wisconsin

The meeting was called to order at 9:40 A. M. by President Flinn. All council members were present except Mr. Weldon. Messrs. Wederbrand, Miller and Presley attended also.

The minutes of the Council Meeting of October 13 were given final approval.

President-Elect Thorstensen reported on the activities of the technical committees. (Mr. Thorstensen will prepare copy on this report and it will become an appendage to these minutes.)

Mr. Thorstensen was requested to ask each committee chairman to review all methods in his group and to submit a recommendation on each method:

(a) to make it official
(b) to continue as provisional
(c) to drop it

This then will be presented to the membership at the business session of the annual meeting for final vote.

It was the consensus of Council that this type of review should be made an annual affair so that methods which are no longer applicable should be dropped from our methods booklet.

Mr. Thorstensen was requested to reconstitute the Mechanical Leather Committee of about five members and to define the scope and character of their program.

Mr. Alfred Hopton was appointed a member of the Physical Test Committee.

The matter of providing literature references on the original publication of data on which methods are based was discussed. Mr. Thorstensen is to ask each committee chairman to provide this information and it is to be published as a section of the methods booklet.

Dr. Stubbings made a motion, which was seconded by Dr. Compton, that, in all future methods development, the literature reference of the data on which the method is based be included in the method.

Council considered the proposal of the Waverly Press on costs of publishing the Journal. The matter was referred to the new Editor for his consideration.

A paper by Dr. Baumann, presented at the International Union of Leather Chemists Societies' Meeting, was approved for publication in the Journal.

A paper by Dr. Schultz is to be re-typed by Mr. Thorstensen's office and then this paper is to be presented to the new Editor for consideration of publication.

Mr. Harold R. Miller, chairman of the Convention Committee, reported on the composition of his committee and submitted a budget as follows:

Members of Committee as of January 13, 1956

Chairman	H. R. Miller
Vice Chairman	C. E. Retzsch
Hotel & Transportation	L. E. Meyers
Entertainment	J. H. Stumpf
Golf	H. R. Willis and D. C. Bundy
Ladies	Not yet obtained
Publicity	Not yet obtained

Proposed Agenda

Sunday—June 17

Committee Meetings
Council Meeting
President's Reception

Monday—June 18

A.M.—President's address and four technical papers
P.M.—Golf Tournament (weather permitting)
Evening—Dinner-dance, with awarding of golf prizes

Tuesday—June 19

A.M.—Four technical papers
P.M.—Five technical papers
Evening—Banquet, installation of new officers

Wednesday—June 20

A.M. and P.M.—Practical leather making symposium closing at 4:00 P. M. sharp

PROPOSED BUDGET

Receipts:		
Member Registration @$6.00	(270)	$1,620.00
Guest Registration $@12.00	(40)	480.00
Display Rental		900.00
Total Receipts		$3,000.00
Expenditures:		
Registration Expenses		$ 350.00
Transcription		600.00
Slides, Public Address, etc.		200.00
Social and Entertainment		1,200.00
Golf Tournament		350.00
Ladies Activities		300.00
Total Expenditures		$3,000.00

This report and budget was approved by Council.

It was suggested that Mr. Meyers, who has charge of hotel arrangements, be asked to provide a schedule of transportation of the facilities available for going to the annual meeting. Also that someone of the committee be designated to handle publicity.

Mr. Wederbrand reported he had obtained seven subscriptions for booths; two of which wish only to contribute but not to have a display. He has several others pending decisions. Each council member was asked to submit to Mr. Wederbrand a list of any potential displayers that they might think of.

Mr. Wederbrand presented a budget for Council's approval for the year of 1956. With small changes as now incoporated in the following copy, this budget was approved by Council.

	1955 Proposed	1955 Actual	1956 Proposed
GENERAL ACCOUNT: INCOME			
Dues	$10,750.00	$11,177.91	$11,500.00
Interest on United States Bonds	1,100.00	1,037.91	1,100.00
By-Laws and Methods	500.00	290.10	100.00
Annual Meeting	—0—	—0—	—0—
Directory	850.00	807.00	800.00
Total	$13,200.00	$13,312.92	$13,500.00
GENERAL ACCOUNT: EXPENSE			
Executive Secretary's Expense	$ 4,200.00	$ 4,200.00	$ 4,200.00
Secretary's Office Expense	1,000.00	835.38	1,000.00
Annual Meeting	200.00	449.14	400.00
Committee Work	100.00	81.00	100.00
Council Meeting	100.00	54.50	100.00
Audit	150.00	175.00	175.00
Printing of Methods and By-Laws	500.00	1,535.99	500.00
Printing Directory	850.00	888.23	900.00
Reserve for Uncollectible Account	300.00	293.50	300.00
Winheim Award	100.00	100.00	—0—
Total	$ 7,500.00	$ 8,612.74	$ 7,675.00
Surplus on General Accounts	$ 5,700.00	$ 4,700.18	$ 5,825.00
JOURNAL ACCOUNT: INCOME			
Advertisements	$12,000.00	$12,143.70*	$12,000.00
Subscriptions	5,000.00	5,208.81	5,200.00
Journals, Volumes, Reprints	2,000.00	2,378.32	2,400.00
Dues, S.L.T.C., Difference	950.00	957.50	950.00
Total	$19,950.00	$20,688.33	$20,550.00
JOURNAL ACCOUNT: EXPENSE			
Printing and Publishing	$15,000.00	$15,447.57	$17,000.00
Printing Reprints	2,200.00	2,088.20	2,400.00
Copyright, Insurance, Storage	500.00	495.05	500.00
Abstracts and Translations	400.00	249.95	400.00
Editor Compensation	1,000.00	1,500.00	3,600.00
Editor Office Expense	700.00	463.94	700.00
Advertising Discount Allowed	150.00	127.65	150.00
Total	$19,950.00	$20,372.36	$24,750.00
Surplus on Journal Account	—0—	315.97	—4,200.00
Surplus	$ 5,700.00	$ 5,016.15	$ 1,625.00

*Estimated December advertising

Mr. Maeser, chairman of the A.S.T.M.—A.I.C.A. Committee, made a report on the development of this committee and the composition of the committee. This report was accepted by Council and ordered published in the Journal.

The Perkin's Centennial program was briefly reported by Dr. Compton and approved by Council.

Mr. Meo, reporting for the committee on the selection of a new editor, reported that they wished to offer a new concept in the matter of editorship of the Journal; namely, to have an editor-in-chief and to employ a managing editor for the actual work. After considerable discussion, on a motion by Mr. Thorstensen and seconded by Dr. Stubbings, it was unanimously agreed to accept the concept as reported by the committee. On a motion made by Dr. Stubbings and seconded by Mr. Meo, it was unanimously approved that the editor-in-chief be appointed for a three year period, and that he cannot be re-appointed to succeed himself.

Then to summarize the entire situation Dr. Flinn outlined the following proposal:

1. That the assignment of editor-in-chief be offered to Dr. Henry Merrill of the B. D. Eisendrath Memorial Laboratory.
2. That the assignment of managing editor be offered to Dr. Watt of the Lowell Technological Institute.
3. That Mr. Meo and his committee will make the final arrangements, and they are to include the following in written record:
   a. the arrangement is on a one year trial basis with the understanding that if everything is satisfactory it will be so continued.
   b. a written agreement by the president of the Lowell Technological Institute be obtained indicating that the arrangements being made are agreeable to the Lowell organization.
   c. the printing of the Journal be continued at the Easton Printing Company until such time as the editor may present information to Council that would indicate a change was desirable.
   d. in the written arrangements to be made, it should be spelled out in detail the remuneration which is to go to the parties concerned and the responsibilities which they are assuming under the agreement. That there must be a coordination between the responsibilities and duties of the editor-in-chief and the managing editor.
   e. when these agreements are in written form they are to be submitted to President Flinn with copies going to the Secretary's office.
   f. maximum amounts of remuneration were mentioned to serve as a guidance to Mr. Meo and his committee in making these negotiations.

The above matter was considered as a motion by Mr. Thorstenson seconded by Mr. Meo, and unanimously approved by all present.

The report of the Nominating Committee's slate of candidates for 1956 was approved unanimously by Council, and will be published in the Journal.

Supplement No. 4 is to be ordered in such quantity as will permit giving a copy to each member and each subscriber and with a small supply in addition. The Secretary is to determine this and arrange for it with the printer.

Dr. Flinn has provided the "Foreword" of the publication and Mr. Twining will arrange having it printed.

A request was received from Dr. Burton for the A.L.C.A. to designate a representative on the International Commission of Leather Chemists Societies for Physical Testing. Council appointed Mr. Maeser to this responsibility.

The following new members were elected to active membership:

A. Kawamura
William J. Langley
Victor Mattei

The following member's status was changed to active:

John F. Burkart

A list of members who had resigned or been dropped by virtue of nonpayment of dues was read and Council was asked to take note of any such members that they felt ought to be further advised.

The following is a list of the deceased members:

Lester Armstrong
W. H. Barrett
Edwin I. Chalmers
James Connelly
Charles W. Lizer
A. Sauer
J. Stodola
D. Williams

Dr. Flinn brought up the matter for discussion as to whether it might be desirable to have some policy in the course of selecting council candidates in the future whereby one such candidate would be a member to represent the interest of the non-United States members of the A.L.C.A. After considerable discussion a study committee was appointed—Dr. Stubbings, chairman members—Dr. Compton and Mr. Weldon. They are to study the matter and submit their recommendations to the Council through the President with a copy to the Secretary.

There being no further business, upon motion duly made and seconded, the meeting adjourned.

Respectfully submitted,

FRED O'FLAHERTY, *Secretary*

REPORT FROM THE TECHNICAL COMMITTEES OF THE A. L. C. A.

The following is a report of the various chairmen of the A.L.C.A. technical committees.

There was no report from the Vegetable Leather Committee.

Mr. E. L. Rayfield, chairman of Vegetable Tannin Analysis Committee, reported that he had received an interim report from Mr. Tom Blair and Mr. Jack Wagoner on their investigation of methods J-10 and A-11 with reference to the proper sampling and preparation of solid extracts, but the work on this is still in progress. Mr. Sverre Dahl has recommended that Mr. Jany's report on the Detection and Estimation of Lignosulfonates be published without the report on the additional cinchonine method determinations which verified Mr. Jany's results.

There was no report on Hide Powder from Mr. Ira Clarke.

The sub-committee on Industrial Wastes reported that they had completed reorganization of the committee as per our suggestions previously given.

Also that they had continued to serve as a clearing house for information pertaining to the treatment of tannery wastes. Six requests for specific information were handled. This report was made by Mr. Jack Wagoner.

Mr. R. S. Adams, chairman of the Mineral Leathers Committee, reported that they are continuing their work on the Lehigh basicity method for leathers. The analysis of the first set of samples (blue stock) has been completed and reported to the members of the committee. The next set of samples in finished side leather has been sent to the members. A deadline of February 29 has been scheduled for this work.

Mr. William Roddy, chairman of the Preparatory and Post-Tanning Process Committee, reported that since completing work on the G Series nothing further has been done. At the same time there was no report from Dr. Koppenhoefer, chairman of the sub-committee on Fats and Oils.

Mr. Meith Maeser, chairman of the Physical Testing Committee, reported that the Water Penetration Sub-Committee have tests being run in one laboratory to screen several methods of automatic end point determination. There are reasons to feel at the present time that the number of cycles for penetration is not a realistic measure of water resistance. Other values may have more meaning.

Mr. Maeser advised that he is preparing a letter ballot to all members of the Physical Testing Committee to determine which methods should be recommended to the Association for adoption as official methods and which should be kept provisional or otherwise disposed of.

In regard to the Standards and Specifications Committee, the only matter to come before this committee was one of duplication between Federal and Military Specifications. Apparently a satisfactory solution will eventually

be reached as indicated in the letter received from Mr. Willis S. MacLeod, Director, Standardization Division. Mr. MacLeod advises that "when Federal Specifications are revised every effort is made by General Services Administration to include requirements of Agency Specifications covering like commodities, making it possible to cancel departmental specifications. Such is the case of Military Specification MIL-L-11636B and Interim Federal Specification KK-L-00191a which is now being coordinated by the Department of Defense." It is expected that the latter will be cancelled.

There was no further report from sub-committees on Fastness of Colored Leather to Light, the committee on Water Penetration on Fastness of Colored Leather to Light, the committee on Water Penetration, nor the committee on Sampling. The Leather Finishes sub-committee reported and recommended publication of Method K-12, Method for Testing Resistance of Colored Leather to Bleeding. A copy of this will be sent to the editor to be printed.

The new committee for the evaluation of Raw Stock held its first meeting on the afternoon of Friday, January 13, at the Plankinton Hotel in Milwaukee.

E. B. THORSTENSEN, *President-Elect*

REPORT OF THE COMMITTEE ON JOINT ACTION WITH THE ASTM

TO THE COUNCIL OF THE A. L. C. A.

January 10, 1956.

At the meeting of the Council on February 11, 1955, the writer was directed to contact officers of the American Society for Testing Materials and discuss the possibility of developing physical testing methods for leather which would be acceptable to both societies. Mr. Hess and Mr. Speight of the ASTM and Ed Thorstensen and the writer met in New York City on April 15, 1955, and discussed possible ways in which the two organizations could work together. The minutes of this meeting were presented to Council at its meeting on May 22, 1955 and are published in the J. A. L. C. A. for Aug. 1955.

At its meeting on May 22, 1955, Council decided that inasmuch as the ALCA had never before established working agreements with other organizations, it was desirous to obtain the approval of the membership before making any such agreements. The matter was presented to the membership at the annual meeting on May 24, 1955. The membership agreed, with only scattered dissenting votes, that the ALCA should undertake cooperative

work with the ASTM in developing methods for the physical testing of leather.

At the Council meeting on May 25, 1955, the writer was requested to set up a working agreement with the ASTM. By correspondence in June and July, 1955, between Mr. Hess and the writer, it was agreed that a small steering committee should meet and establish a permanent organization for conducting such joint efforts. The steering committee met at the ASTM headquarters in Philadelphia on October 12, 1955. The minutes of that meeting are attached to this report. The actions of the steering committee were reported verbally to the Council at its meeting on October 13, 1955. The Council approved of the organization suggested by the steering committee and directed the writer to complete a permanent agreement with the ASTM.

The advisory committee which was set up by the steering committee met at the ASTM headquarters in Philadelphia on December 6, 1955, and effected a permanent organization and listed the names of possible members of working committees. The minutes of the December 6th meeting are attached to this report.

In summary, a joint ALCA-ASTM ten member advisory committee has been established. Joseph R. Kanagy, Chief of the Leather Section, National Bureau of Standards, is Chairman, and Malcolm Battles of A. C. Lawrence Co. is Secretary. One member of the committee is selected by the Council of the ALCA and it is the committee's suggestion that the Council appoint the Chairman of the Physical and Mechanical Properties Committee as this member. One member of the Advisory committee is designated by the ASTM. The other eight members are selected from specific segments of the leather industry as noted in the minutes of the meeting.

The Advisory committee has the responsibility of setting up working committees and suggesting the methods which they should develop. It was agreed by the members of the Advisory committee present at the meeting on December 6th, that the first business of the working committees would be to study the present physical testing methods of the ALCA and from them develop methods which would be satisfactory to both associations.

Methods which are developed by the joint committee will be submitted to each association for approval. When such approval has been given, the methods will be published as joint standards and carry both the ASTM and the ALCA seals.

Respectfully submitted,

MIETH MAESER, *Chairman,*
Physical and Mechanical Properties Comm.

MINUTES OF THE ALCA - ASTM MEETING

A steering committee consisting of three members of the ALCA and three members selected by the ASTM met on October 13, 1955, at the headquarters of the ASTM. The committee consisted of Alfred M. Compton, E. F. Houghton & Co.; R. J. Wirshing, General Motors Corp.; Arthur M. Kay, Howe's Leather Co., Inc.; Malcolm Battles, A. C. Lawrence Co.; Joseph Kanagy, National Bureau of Standards; with M. Maeser, United Shoe Machinery Corp., Chairman. Mr. Frank Speight, Assistant Technical Secretary and Mr. R. E. Hess, Technical Secretary of the ASTM, met with the committee.

The steering committee discussed several possible ways in which a working committee with the responsibility of devising physical testing methods for leather and leather products could be set up. The following plan was finally accepted.

A permanent ten member advisory committee was established. Each member of this committee was chosen to represent a particular segment of the leather industry. The functions of this committee are:

(a) to secure committee members from the various segments of the industry;

(b) to draw up the by-laws of the committee, call its first meeting, effect its organization, and suggest the methods on which it shall work.

The steering committee tentatively set up the following advisory committee:

A.L.C.A.—It is suggested that the chairman of the Physical and Mechanical Properties Committee of the ALCA be a member of this committee.

A.S.T.M.—A member to be designated.

Producers

Light leather—Malcolm Battles, A. C. Lowrence

Heavy Leather—Arthur Kay, Howe's Leather Co.

Mechanical leathers (gaskets, straps, etc.)—to be selected

Users

Shoe Leathers—R. G. Ashcraft, Endicott Johnson

Upholstery and other industrial users—R. G. Wirshing, General Motors

Gloves, bags, garments, straps, etc.—George Meyer, Liberty Dressing Company.

General Interest

Joseph Kanagy, Chief of Leather Section, National Bureau of Standards

Allied Trades

Robert Shaw, Rohm & Haas

The willingness of Mr. Ashcraft and Mr. Meyer to serve will be ascertained by the Chairman. Mr. J. T. Eaton of Houghton & Co. will be requested to designate a member of his staff to serve as the representative of the Mechanical Leathers group.

Each member of the advisory committee will be asked to bring names of men in his segment of the industry who would be satisfactory committee members, to the meeting on December 6, 1955. After the advisory committee is organized, it will select the desired committee members from the lot of names submitted and set a date for the first committee meeting.

It was generally agreed by the members of the steering committee that the present ALCA methods will be considered by the new working committee as its first business. It can be assumed that many of the ALCA methods will require no change in order to be acceptable to the new committee. There are other methods which will undoubtedly be changed rather completely.

At the conclusion of the steering committee meeting, a joint publicity release was prepared by Mr. Speight and the writer. It was tentatively agreed, waiting the approval of the Council of the ALCA, that the Publicity Department of the ASTM should edit the release and send thirty copies to Fred O'Flaherty, Secretary of the ALCA, who will in turn, send it either directly or through Irving Glass of the Tanners' Council to the Leather and Shoe trade journals. The ASTM will send the same release to a selected list of journals and bulletins and will publish it in the ASTM Bulletin.

MIETH MAESER, *Chairman*
ALCA-ASTM Steering Committee

MINUTES OF MEETING OF ALCA – ASTM JOINT COMMITTEE

The committee met on December 6th at ASTM Headquarters in Philadelphia, the meeting being called to order at (9:35 a.m.) by Mr. Maeser acting as Chairman. The following members were present:

Mr. Mieth Maeser—Research Engineer, United Shoe Machinery Corp., Beverly, Mass.

Mr. Ralph J. Wirshing—Head, Chemistry Dept., Research Staff, General Motors Corp., P. O. Box 188, North End Station, Detroit 2, Mich.

Mr. B. L. Lewis—Engineer, Tinius Olsen Testing Machine Co., Easton Road, Willow Grove, Penna.

Mr. J. N. Smith—Manager, Hydraulic & Trans. Dept., E. F. Houghton & Co., 303 West Lehigh Avenue, Philadelphia 33, Penna.

Dr. J. R. Kanagy—Chief of Leather Section, National Bureau of Standards, Washington 25, D. C.

Mr. R. E. Hess—Technical Secretary, A.S.T.M., 1916 Race St., Philadelphia 3, Penna.

Mr. R. G. Ashcraft—Superintendent, Chemical Dept., Endicott-Johnson Corp., Endicott, New York.

Mr. A. N. Kay—Chief Chemist, Howes Leather Co., Inc., West Hickory, Penna.

Mr. G. H. Meyer—President, Liberty Dressing Co., Inc., Gloversville, New York.

Mr. F. Y. Speight—Asst. Sec., A.S.T.M., 1916 Race St., Philadelphia 3, Pa

Absent were:

Mr. M. H. Battles—A. C. Lawrence Leather Co., Peabody, Massachusetts.

Mr. R. Shaw—Rohm & Haas Co., 5000 Richmond Street, Philadelphia 37, Penna.

For the benefit of those members who were not present at the first meeting, Mr. Maeser briefly presented the reasons for this committee's being established, and indicated that it had been agreed to work on Physical Test Methods first and to leave consideration of Chemical Methods to a later date.

The first item of business was the election of officers. Dr. Kanagy nominated Mr. Maeser for chairman but the latter felt compelled to decline due to pressure of other business. Mr. Ashcraft nominated Dr. Kanagy, this was seconded by Mr. Lewis and approved unanimously.

Dr. Kanagy then took the chair. Mr. Wirshing was nominated for Vice-chairman, seconded by Mr. Ashcraft and approved unanimously.

Mr. Maeser nominated Mr. Battles for secretary, seconded by Mr. Kay and approved unanimously. In the absence of Mr. Battles, the chairman appointed Mr. Kay to act as secretary for the meeting.

At the last meeting, members had been asked to consider the names of people and companies who might be interested in acting on the main committee. The following were suggested:

General Interest:

Baldwin, Dr. M. M., Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio.

Beal, Mr. G. D., Mellon Institute, 4400 Fifth Avenue, Pittsburgh 13, Pennsylvania.

Chouinard, Dr. A. E., Lowell Technological Institute, Department of Leather Engineering, Lowell, Massachusetts.

Naghski, Dr. Joseph, Eastern Utilization Research Branch, U. S. Department of Agriculture, 600 Mermaid Lane, Philadelphia 18, Pennsylvania.

O'Flaherty, Dr. Fred, Tanners' Council Research Laboratory, University of Cincinnati, Cincinati 21, Ohio.

Orthmann, Mr. A. C., The Orthmann Laboratories, Inc., 407 E. Michigan Street, Suite 312, Milwaukee 2, Wisconsin.

Stubbings, Prof. Robert, Division of Leather Technology, Lehigh University, Bethlehem, Pennsylvania.

von Fuchs, Dr. George, 1221 Garland Avenue, Niagara Falls, New York.

Walker, Mr. H. B., 1311 Frog Hollow Road, Box 60, Rydal, Pennsylvania.

Producers:

Artz, Mr. F. E., Howes Leather Company, Inc., 310 Summer Street, Boston 10, Massachusetts.

Barth, Mr. A., Chief Chemist, Chicago Rawhide Manufacturing Company, 1301 Elston Avenue, Chicago 22, Illinois.

Battles, Mr. M. H., Director of Research, A. C. Lawrence Leather Company, Peabody, Massachusetts.

Butz, Mr. G. A., Endicott Johnson Corporation, Endicott, New York.

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Cox, Mr. W. R., Seton Leather Company, 62 Verona Avenue, Newark, New Jersey.

Doherty, Mr. E. E., Bona Allen, Inc., Buford, Georgia.

Downing, Mr. G. V., Leas and McVitty, Inc., Salem, Virginia.

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Hatton, Mr. J. B., Eagle-Ottawa Leather Company, Grand Haven, Mich.

Kevil, Mr. Gerard, International Shoe Company, 1509 Washington Avenue, St. Louis, Missouri.

Knisely, Mr. C. K., Tanners' Research Corporation of New Jersey, 410 Frelinghuysen Avenue, Newark, New Jersey.

Kremen, Dr. S. S., Leather Research Corporation, P. O. Box 153, Cincinnati 14, Ohio.

McDonnell, Mr. A. E., Endicott Johnson Corporation, Endicott, New York.

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Stout, Mr. F. Sturgis, John R. Evans and Company, Second and Erie Street, Camden, New Jersey.

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Wagoner, Mr. J. F., Armour Leather Company, 500 Arch Street, Williamsport, Pennsylvania.

Williams, Mr. D., International Packing Division, Graton and Knight Company, 356 Franklin Street, Worcester 4, Massachusetts.

Wilson, Mr. C. David, Fred Rueping Leather Company, Fond du Lac, Wisconsin.

Consumers:

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Beckerlegg, Mr. H. V., Chief Chemist, Fisher Body Division, General Motors Corporation, Detroit, Michigan.

Burke, Mr. R. S., Manager, Merchandising Development and Testing Laboratory, Dept. 817, Sears Roebuck and Company, 925 South Homan Avenue, Chicago 7, Illinois.

Byron, Mr. J. W., General Shoe Corporation, Seventh Avenue, Nashville 3, Tennessee.

Collins, Mr. F. Leslie, International Shoe Company, 1820 Cherokee Street, St. Louis, Missouri.

Fitzgerald, Mr., Director of Research, Draper Corporation, Hopedale, Massachusetts.

Jaynes, C. Elmer, Address correspondence to:- Commanding Officer, WMAC, Attention: C. Elmer Jaynes, Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio.

Leithauser, Mr. Gene, Research Staff, General Motors Corporation, Box 188, North End Station, Detroit 2, Michigan.

Mann, Mr. C. W., QM Research and Development Center Operations, Quartermaster Research and Development Center, Natick, Massachusetts.

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Allied Trades:

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Black, Dr. C. K., Director of Technical Laboratories, Box 386, Wilmington, Delaware.

Casnocha, Mr. J. A., Sandoz Chemical Works, Inc., 61 Van Dam Street, New York, New York.

Davis, Mr. R. B., Marshall Laboratory, E. I. du Pont de Nemours and Company, Inc., 3500 Graysferry Road, Philadelphia 45, Pennsylvania.

Dudley, Mr. C. F., Jr., Dow Corning Corporation, Midland, Michigan.

Frodin, Mr. E. E., Chicago Rawhide Manufacturing Company, 1301 Elston Avenue, Chicago 22, Illinois.

Hilterhaus, Mr. Fred, Newark Leather Finish Company, 8 Wilhelm Street, Harrison, New Jersey.

Kirk, Dr. J. Stanley, General Dyestuff Corporation, Division of General Aniline and Film Corporation, Rensselaer, New York.

Koppenhoefer, Dr. R. M., Socony-Mobil Oil Company, 412 Greenpoint Avenue, Brooklyn 22, New York.

Leavey, Mr. Joseph, E. I. du Pont de Nemours and Company, Inc., Wilmington, Delaware.

Meo, Mr. Dominic, Salem Oil and Grease Company, Salem, Massachusetts.

Nason, Mr. H., Monsanto Chemical Company, St. Louis 1, Missouri.

Persons, Mr. Francis, Koppers Company, Inc., Pittsburgh 19, Pennsylvania.

Quarck, Mr. Rolf, Chemtan Company, Port Washington, New York.

Radi, Mr. L. J., RBH Dispersions Division, Interchemical Corporation, Bound Brook, New Jersey.

Retzsch, Mr. C. E., Nopco Chemical Company, Harrison, New Jersey.

Rohm and Haas Company, 222 West Washington Square, Philadelphia 5, Pennsylvania.

Wint, Mr. R., Hercules Powder Company, Wilmington 99, Delaware.

Wismer, Dr., Ciba Products Corporation, Kimberton, Pennsylvania.

Liason:

A.S.T.M. Group headed by:

Thorn, Mr. F. C., Director of Research, Garlock Packing Company, Palmyra, New York.

In addition to the above, it was agreed that the chairman would get in touch with Dr. O'Flaherty for the names of other persons who might be interested.

By-Laws and Scope

Mr. Speight handed out copies of the mimeograph of Suggested BY-LAWS to those members who were not present at the first meeting. After some discussion, Messrs. Maeser, Wirshing and Ashcraft were appointed as a

committee to draw up suitable by-laws, following the lines of the mimeograph, for consideration by the committee.

Discussion followed as to what subcommittees should be appointed. It was finally agreed to establish five major subcommittees as follows:

(1) Nomenclature and Definitions.
(2) Sampling and Conditioning for Physical Testing.
(3) General Physical Testing.
(4) Research.
(5) Editorial.

Subcommittee No. 3 was to consider methods which were applicable to all types of leather, but it was tentatively agreed that this subcommittee should have two immediate sections stemming from it:

Section 1—Physical Testing of Heavy Leather.

Section 2—Physical Testing of Light Leather.

Considerable discussion followed as to whether Sections 1 and 2 of subcommittee No. 3 should be subdivided according to type of method, or type of leather, or to groups of either methods or leathers. It was finally agreed to wait until the main committee had been established and the matter could be discussed more clearly in the light of personnel available and their interest and equipment. In order to save time, it was agreed that, when the chairman circularized the names listed above (together with any others reaching him from members of this committee, from correspondence with Dr. O'Flaherty, or otherwise) he would indicate that it was proposed to use the present, published, methods of the ALCA as a basis for immediate study.

Mr. Hess said that the ASTM would be glad to help with the necessary mimeographing and he felt that the circular should indicate the scope of the committee's present intent, viz., The Improvement and Development of Methods for the Physical Testing of Leather. Both suggestions were approved unanimously.

No date was set for the next meeting, as it was realized that this would depend on the results of the circular letter. It was agreed that the chairman should arrange for the next meeting as soon as he felt it possible to do so, preferably within the next two months.

The Acting Secretary was asked to type the minutes of this meeting and submit copies to all members as soon as possible.

There being no further business, the meeting adjourned at 12:30 p. m.

Respectfully submitted,

ARTHUR N. KAY, *Acting Secretary.*

Tanning Studies with Epoxy Resins*

By E. M. Filachione and E. H. Harris, Jr.

*Eastern Regional Research Laboratory***
Philadelphia 18, Pennsylvania

Although the chemical properties of 1,2 epoxides have been well established for some time, it is only recently that the interaction of this class of organic compounds with proteins has been reported. In 1944 Fraenkel-Conrat [1] reported the reaction of ethylene oxide, propylene oxide, and epichlorohydrin with various water soluble proteins. Interaction was readily accomplished under mild conditions and in aqueous solution. The isoelectric point of the treated proteins was increased by 1 to 3 pH units, indicating that the carboxyl groups were largely esterfied. Amino, phenolic, and sulfhydryl groups also appeared to participate in this reaction.

Fearnley and Speakman [2] investigated the action of diepoxybutane on wool and found that supercontraction of wool was prevented, presumably because of formation of new cross linkages between carboxyl groups of peptide chains. In contrast Alexander el al. [3] observed that only epichlorohydrin esterified the carboxyl groups of wool to any considerable extent.

Sykes [4] investigated the reaction of propylene oxide, epichlorohydrin, and vinyl cyclohexene dioxide with sheepskin collagen. The monofunctional propylene oxide had little effect on the structural stability of collagen. In alkaline solution epichlorohydrin caused an increase in shrinkage temperature (Ts) to about 78° C. (after three to seven days) comparable to aldehyde tannage. The diepoxy compound, vinyl cyclohexene dioxide, tanned collagen over a wide range of pH. Although the shrinkage temperature (72°C) was not very high the bonds introduced were stated to be exceptionally stable.

Immendorfer [5] studied the tanning action of various other epoxides. It was reported that firm, full, supple and well tanned leathers were obtained upon treatment of hide or calfskin with 15–20% by weight of butadiene monoxide, butadiene dioxide, butanediol diglycidyl ether, phenoxybutene oxide and epichlorohydrin.

Within recent years the epoxy resins, a new class of condensation polymers, have become commercially available. In general these are condensation products of epichlorohydrin with bisphenol [6–11]. The commercial resins are mixtures of polymers with molecular weights in the range of about 400 to 8000 [7]. An important structural feature of these epoxy resins is the presence of terminal glycidyl ether groups, so that they can be considered as diepoxides. Hence it was of interest to investigate the behavior of the epoxy resins toward

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**A laboratory of the Eastern Utilization Research Branch, Agricultural Research Service, United States Department of Agriculture.

hide substance. Exploratory work indicated that one of the epoxy resins was particularly suitable for treatment of hides in aqueous system. This paper is a preliminary report on tanning with this resin in aqeous suspension.

Experimental

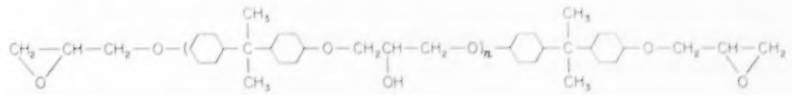
Materials. Limed cowhides and pickled calfskins were obtained from local tanneries. The cowhides were thoroughly rinsed and then pickled in a hydrochloric acid—sodium chloride pickle. The pickled raw materials were stored in a cold room at 36° F. Before tanning, the hides and skins were depickled by treating for 5 to 6 hours with sodium bicarbonate solution (pH 6.5), washed in water for 24 hours, treated for 7 to 8 hours with sodium acetate—acetic acid buffer (pH 4.9), washed in water for 24 hours, then dehydrated with acetone.

The epoxy resin used in this study was Epon 562.***

Treatment with Epoxy Resin. Small strips, approximately 1 x 3 inches, were cut from the acetone dehydrated cowhide or calfskin. About 10 g. of the cowhide and 6.5 – 8.0 g. of calfskin were used in the various treatments with the epoxy resin. These samples were wet back with water and generally 100 ml. of water or 0.2M buffer solution, 10 g. of various salts, and 5 g. of Epon 562 were added. The reaction was carried out at room temperature in closed bottles which were gently agitated by rolling on a low speed jar mill. Specimens, ¼ x 2-⅜ inches were periodically removed, rinsed thoroughly, and the shrinkage temperature (Ts) determined. The Ts was determined on freely suspended specimens mounted in a holder accommodating four samples. A few spot checks indicated that the Ts agreed reasonably well with the values obtained using the Theis shrinkage meter. After shrinkage the specimens were immersed in cold water to observe reversibility of shrinkage. After six days of treatment the remaining sample was thoroughly washed in water, allowed to air dry, and examined for leather-like appearance. The data are summarized in Tables I and II.

Discussion

The epoxy resins, which have recently become available, are in general condensation products of epichlorohydrin and bisphenol -A and the following structure has been postulated[6–11].



***Obtained from Shell Chemical Corp., 500 Fifth Ave., New York 36, N. Y. The mention of specific brands or companies is not to be construed as an endorsement by the United States Department of Agriculture of these brands or companies over those not mentioned.

TABLE I

Tanning Cowhide with Epon 562[a]

Exp. No.	Buffer	Salt	pH[b]	Shrinkage Temperature, °C.[c]				
				8 hours	1 day	2 days	3 days	6 days
1	Lactic acid, 0.2M	None	2.5–2.6	59	55	59	61	57
2	" "	NaCl	4.2–8.7	58	64	65	69	67
3	" "	Na_2SO_4	3.7–4.2	55	61	60	62	66
4	" "	$MgSO_4.7H_2O$	3.2–3.5	55	56	58	63	58
5	Sodium lactate, 0.2M	None	6.4–7.8	65	71	75[d]	77[d]	77[d]
6	" "	NaCl	10.0–9.6	72	78	80[e]	81	80[e]
7	" "	Na_2SO_4	8.6–9.1	68	77	78	82[e]	82[e]
8	" "	$MgSO_4.7H_2O$	7.8–8.6	66	68	74[d]	78[d]	77[e]
9	Water	None	5.8–7.3	66	69	77[d]	80[d]	79[d]
10	"	NaCl	10.0–9.8	69	76	77	81	79[e]
11	"	Na_2SO_4	8.6–9.6	67	74	77[d]	81[e]	82[e]
12	"	$MgSO_4.7H_2O$	8.1–8.8	63	67	70	76[d]	76[e]
13	$NaHCO_3$, 0.2M	None	8.1–8.3	68	74	75[d]	77[d]	76[d]
14	"	NaCl	9.0–9.2	70	74	79[d]	79[d]	77
15	"	Na_2SO_4	8.2–8.7	70	77	78[d]	81[e]	80[d]
16	"	$MgSO_4.7H_2O$	7.6–7.8	69	72	73	75	75[d]
17	Na_2CO_3, 0.2M	Na_2SO_4	10.0–9.8	74	80[d]	78[d]	80[d]	83[e]
18	$Ca(OH)_2$, saturated	None	9.1–9.0	78[e]	80[d]	80[d]	81	80[d]
19	"	Na_2SO_4(2.5g)	11.2–10.8	73	75	—	78[d]	78[d,f]
20	"	Na_2SO_4(5 g)	10.7–11.0	75	76	—	82[e]	82[e,f]
21	"	Na_2SO_4	10.8–11.1	79[d]	78[d]	—	86[e]	86[e,f]
22	"	NaCl	10.3	77[d]	79[d]	77[d]	79[d]	79[e]
23	"	$MgSO_4.7H_2O$	9.1–9.2	64	68	84[e]	77[d]	77
24	$Ca(OH)_2$, 1.0 g.	Na_2SO_4	12.0–11.8	68	71	85[e]	85[e]	84[e]
25	MgO, 1.0 g.	None	10.2–9.9	64	70	—	79	82[d,f]
26	"	Na_2SO_4	10.4–10.2	66	75	—	81[e]	83[e]

a. Hide (Ts=60°C) generally equilibrated overnight with buffer or buffer salt solution before adding Epon 562. Ten grams of salt were present in the aq. solution unless otherwise noted.
b. Range for the 8 hour to 6 day interval.
c. Samples washed for about 1 day in tap water.
d. Ts specimens showed good recovery of length (Ca 90–96%) on cooling.
e. Ts specimens showed excellent recovery of length (96–100%) on cooling.
f. Treatment carried out for 5 days.

Epon 562, one of the epoxy resins, is stated to be an aliphatic modification [6]; a condensate of glycerol (rather than bisphenol) with epichlorohydrin [1,2]. In both types of resin the terminal groups of the chain are probably glycidyl ether groups and hence may be considered examples of commercially available diepoxides. Epon 562 is a moderately viscous liquid (viscosity C to F on the Gardner-Holdt Scale) having an equivalent weight (as an epoxide) of 140–165. It apparently is of low average molecular weight probably about 300, and slightly soluble in water.

It was found that Epon 562 reacted under mild conditions and in aqeous suspension to effect a tanning action on cowhide and calfskin. The tanning action was accompanied by an elevation of the shrinkage temperature and

TABLE II

Tanning Calfskin with Epon 562[a]

Exp. No.	Buffer	Salt	pH[b]	Shrinkage Temperature ° C.[c] 8 hours	1 day	2 days	3 days	6 days
1	Lactic acid, 0.2M	None	2.4	58	60	59	55	59
2	" "	Na_2SO_4	3.1–3.7	52	57	57	64	61
3	Sodium lactate, 0.2M	None	6.6–7.9	64	68	73	74	77[e]
4	" "	NaCl	10.0	68	79	80	81[d]	80[d]
5	" "	Na_2SO_4	7.8–9.6	64	71	78	82[d]	82[d]
6	Water	None	5.7–7.0	65	65	73	74	78[e]
7	"	NaCl	10.0	66	76	78	78	78
8	"	Na_2SO_4	7.9–10.7	64	70	76	81	83[e]
9	$NaHCO_3$, 0.2M	None	8.6–8.5	68	70	73	74	74[d]
10	"	NaCl	8.7–9.1	66	69	73	75	76
11	"	Na_2SO_4	7.9–8.6	66	73	75	79	81[d]
12	Na_2CO_3, 0.2M	None	10.5	72	77	75	79	77[d]
13	"	NaCl	10.2–10.0	79[e]	82[e]	83[d]	83[e]	82[d]
14	"	Na_2SO_4	10.2	81[d]	85[e]	85[d]	87[e]	86[e]
15	$Ca(OH)_2$, saturated	None	9.9–9.8	77	79	79[d]	75	80
16	"	Na_2SO_4 (2.5 g)	10.0–11.1	73	75	—	79	79[f]
17	"	Na_2SO_4 (5 g)	10.1–11.3	78	80	—	82[d]	82[d,f]
18	"	Na_2SO_4	10.2–11.3	85[d,g]	87[e]	—	88[e]	89[e,f]
19	"	NaCl	10.6–10.8	77[d]	78[d]	80[d]	82[e]	80[d]
20	MgO, 1 g	None	10.0–9.9	70	78	—	82[d]	82[e,f]
21	"	Na_2SO_4	10.1–10.3	78[d]	85[e]	—	85[e]	85[e,f]

a. Calfskin (Ts=60°C) generally equilibrated overnight with buffer or buffer salt solutions before adding Epon 562. Ten grams of salt were present in the aqueous solution unless otherwise noted.
b. Range for the 8 hour to 6 day interval.
c. Samples washed for about 1 day in tap water.
d. Ts specimens showed good recovery of length (Ca 90–96%) on cooling.
e. Ts specimens showed excellent recovery of length (96–100%) on cooling.
f. Treatment carried out for 5 days.
g. Ts after 4 hours was 82° C. with excellent recovery of length on cooling.

reversible shrinkage. These two properties were of value in following the course of the reaction. Little if any tanning effect or reaction appeared to occur at pH below about 6. In the more alkaline range, pH above about 9, however, reaction appeared to be effected in a relatively short time. Tanning, as judged by the shrinkage temperature and reversibility of shrinkage, was effected in a period of from 8 hours to 6 days depending on the conditions. Sodium carbonate, magnesium oxide, or lime water, used with sodium sulfate, appeared to be the most desirable medium for effecting reaction or tanning, at least as regards the rate at which maximum Ts and reversible shrinkage are attained. In general the shrinkage temperature was elevated to approximately 80–85° C. Usually good reversibility of shrinkage was not observed until the Ts reached about 80° C. A sample of the leather prepared as in Experiment 21, Table I (6 days of treatment) was submitted to five successive shrinkages in a Theis meter. The specimen regained its original length on

cooling each time. The Ts values were 83, 80, 79, 79, and 78° C., successively. A sample of formaldehyde tanned material, which also is known to shrink reversibly, showed the following successive Ts values: 89, 75, 69, 67, and 61° C. It is apparent that tanning with Epon 562 results in bonds, probably cross-linked, having a high degree of stability. The leather, after repeated shrinkage, was still flexible and leather-like when dried. When a wet back piece of Epon 562 tanned cattlehide (approximately two inches square) was immersed in boiling water for one minute it shrunk to about 75% of its original area, and regained its original area when placed in cold water.

Particularly in the alkaline pH range, using sodium carbonate, lime water, and magnesium oxide to control pH, the use of inorganic salts such as sodium sulfate resulted in more rapid reaction and a product with better leather characteristics. Sodium sulfate was considerably more effective than sodium chloride or magnesium sulfate probably because the former salt functions as a more active swelling depressant in alkaline solutions[13]. An aqeous sodium sulfate solution which was about 10% by weight with respect to the salt appeared to give the best results. Sodium chloride is probably not a suitable salt to use in this treatment because of the known tendency of epoxides to add hydrogen chloride even from neutral solutions[14]. When sodium carbonate, magnesium oxide, or lime water were used to regulate the pH, along with sodium sulfate, tanning with Epon 562 resulted in suitable leather products from cowhide in one to three days and from calfskin in less than one day.

Summary

Epon 562, an epoxy resin which is commercially available, was found to interact with cowhide and calfskin in aqeous systems to produce a tanning effect. Little if any reaction or tanning appeared to take place in the pH range below about six. High pH seemed most suitable for this reaction as regards the rate at which maximum Ts and reversibility of shrinkage are attained. Treatment with Epon 562 in sodium carbonate, magnesium oxide, or lime water in the presence of sodium sulfate gave the best results. Suitable leather products were obtained from cowhide in one to three days (Ts 80–85° C.) and from calfskin in less than one day (Ts about 85° C.). The leather exhibited the unusual property of reversible shrinkage with no visible damage to leather appearance after shrinkage.

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Received August 21, 1955.

Discussion

Dr. Wallace Windus (Tanimex Corporation): Dr. Filachione and his associates have done an interesting piece of work. It is striking how the conditions resemble the classical method of tanning with formaldehyde—both being on the alkaline side to best advantage, and also the reversible shrinkage is similar.

This suggests that possibly the mechanism of the two tannages are closely related, if not the same. I think it is gratifying that possibly in this case we will have a reversal of the usual procedure of defining the mechanism of tanning before a complete evaluation from a practical standpoint. Perhaps the scientist is finally catching up with the art.

I have only one comment in connection with physical properties being somewhat poorer in some instances. As we all know this depends to some extent, perhaps a large extent, on the amount of tanning material that is used.

Conventional tanning materials if used in excess, produce over-tanning and lower the physical data. In this case approximately 20 to 25 per cent of the epon resin was used on a wet basis. This may be more than is necessary, providing the shrink test will be satisfactory with a smaller amount of material. If less can be used, I think the physical data will be improved. I think there are perhaps a number of questions. Mr. Lotz will now comment.

Mr. W. Robert Lotz (Albert Trostel and Sons): I think the very fact that our author has defined his paper as being in the nature of a progress report sets the pace for the discussion which must follow. Have we questions from the floor?

Dr. Ludwig Seligsberger (Quartermaster Research & Development Center): I just wonder whether you have determined the pH of the leather after washing because I think, maybe you have a little too high a pH.

DR. FILACHIONE: It takes a long time to wash alkali out of the samples. We usually wash these for a period of twenty-four hours; but before running the titration curve the ground-up specimens were electro-dialyzed, so we feel that this particular sample was free of all the inorganic ions that might be absorbed.

MR. FRANK CHADWICK (Atlas Refinery): There has been comment that the epoxy resins are highly toxic. Can you discuss this point from a production level?

FILACHIONE: I am afraid I cannot. I have not asked the suppliers of the resins for any specific information on toxicity. The label on Epon 562 carried the following caution: "this material should not be taken internally. Avoid prolonged contact with skin".

DR. WILLIAM J. MCDERMOTT (Rohm & Haas): Regarding toxicity, aren't these materials oil soluble? Wouldn't they be absorbed into the skin and since they are highly reactive, would you not expect them to react with the protein in the blood and also probably with the skin, itself? So you might end up with a dermatitis of some type. I would suspect that because of their reactivity with proteins that they would be toxic, especially if they are oil soluble.

FILACHIONE: They are soluble in organic solvents.

MCDERMOTT: They do have oil solubility?

FILACHIONE: Probably not in hydrocarbon oils.

MCDERMOTT: I was thinking in terms of solubility in animal oils.

FILACHIONE: Preferred solvents are ketones, esters, and ether alcohols. They may have some solubility in vegetable or animal oils.

MCDERMOTT: My thought was that solubility in animal oils would permit absorption by the skin and reactivity with body proteins could result in a toxic effect. One other question: how about the light stability of the Epoxy resin-tanned leather?

FILACHIONE: I would think the stability would be exceptionally good. I think there are co-valent bonds introduced into the protein by treatment with the epoxides.

MCDERMOTT: Such tanning materials would have added interest, if they gave light-fast leather, providing it is possible to improve them sufficiently so as to give stronger leather.

FILACHIONE: Yes I believe there is this possibility.

MCDERMOTT: It has been mentioned that the physical weakness of Epoxy Resin-tanned leather might be due to the incomplete removal of alkaline

materials. In the process of deliming you treat the raw stock with ammonium chloride, or similar salt as a part of the bating procedure. Could not such salts be used in the deliming or de-alkalizing of such leather to decide whether the weakness is due to incomplete alkali-removal or is inherent in the tannage.

FILACHIONE: You mean, use a deliming operation before testing?

MCDERMOTT: Yes, to make sure you have removed all of the alkaline material.

FILACHIONE: That may be what gives us these results. We would like to repeat some of these experiments and we will look into it.

MCDERMOTT: You use a straight wash?

FILACHIONE: We just wash with water for a day or so.

LOTZ: The question of the solubility in solvents brings to mind the use of the solvent system itself. Would you care to discuss what you think of the use of these epon resins in solvent systems?

FILACHIONE: We have done a little on solvent systems, but we thought it would be more desirable to use a water system. The reaction does not occur as rapidly in the presence of solvent. We have used this resin as well as some of the other epoxy resins in fifty per cent acetone water systems and the shrink temperature did not get as high and the reaction seemed slower.

I think we let the reaction go on for about twelve days in the solvent system, whereas here we considered that we were finished before six days.

DR. STUBBINGS (Lehigh University): Have you tried the epoxy resin as a retannage of a chromium tanned material?

FILACHIONE: We have tanned these epoxy tanned materials with both vegetable and chrome in a qualitative way. They do take up a chromium or vegetable tanning material.

STUBBINGS: Have you tried the reverse—tanning with the epoxy material on top of a chromed tanned leather?

FILACHIONE: We did, but it did not seem to be very suitable because we preferred to treat with this resin in an alkaline medium and we did not get anything that looked too good.

MR. ANDREW J. GARD (The Dow Chemical Company): Did you run the determination on chrome?

FILACHIONE: We did not. We did run shrink temperature of the vegetable retanned material.

GARD: Was there a change that might indicate a stripping of the resin?

FILACHIONE: No, there was an increase in the shrink temperature when we retreated with vegetable—about five to ten degrees.

MR. FRANK EDMONDS (American Cyanamid Company): On the question of retanning with the vegetable, did you drop the pH of that leather with acid so that you could vegetable tan?

FILACHIONE: I think we generally used pH's of around 5—the natural pH of the extract, which was sulphited quebracho and chestnut. It was 5, or a little below that.

MR. W. O. DAWSON (Chemtan Company): I would like to state this: these products are notoriously water-in-soluble. Also Epon 562, being the lowest molecular weight product of the group, is the most water-soluble of this group of compounds sold by Shell. Did you determine the efficiency of the Epon 562? Secondly, have you investigated or inquired from Shell whether or not there may be methods of solubilizing the Epon 562 so that it can be used in a water system without resorting to the use of an emulsifying agent or suspending agent which system you used in your experiments?

FILACHIONE: To answer the first part, we generally just standardized on using a weight of Epon 562 which was 50 per cent of the acetone dehydrated hide material. And we did not try to determine the lowest quantity of Epon 562, which we could use to treat the protein. But I am sure it is considerably below the fifty per cent figure that we used.

Often in our runs we would see the insoluble layer of presumably unreacted Epon 562 at the end of the six-day period. And we generally tested the solution qualitatively for epoxide and there was always epoxide present at the end of the six days.

There may be some side reaction and I think the most important one probably would be hydrolysis of the epoxide by the water. But that should be fairly slow.

In all of these other treatments of proteins with epoxide they generally carry out the reaction for six or seven days and apparently the rate of hydrolysis is slow. In actual commerce, when they want to hydrolyze epoxides they generally use acid catalysts for increasing the rate of hydrolysis of the oxide ring.

There was another part to your question: we have not asked Shell or have not tried to obtain any information on whether these resins can be solubilized. We used the straight suspension in water and the emulsifying agent in water, and you would expect the emulsifying agent to increase the solubility, but it appeared to behave similarly to the unemulsified system, so that we assumed that, if it did increase the solubility, it did not increase it enough to alter the rate of the reaction.

MR. LOTZ: If there are no more questions it is papers like this that represent the building blocks for making new tannages. As a practical leather man I appreciate that this is only the beginning. It is in the nature of a progress report. At the same time, work done here can be expanded into things which can be turned into dollars and cents by the tanneries.

I want to thank Mr. Filachione for his paper and for the presentation. I am sure if you have further questions he will be glad to answer them after the morning session.

The Chemistry of Vegetable Tannins

XIV WATTLE

By ROBERT C. PUTNAM

The structure of wattle tannin has intrigued investigators for a long time. The present article is a summary of the work performed by the author and his colleagues which was reported in part in prior articles of this series. It does not touch upon the work performed by others in this field. This work was carried out somewhat as a side issue since the main tannin under investigation at the time was quebracho.

PURIFICATION

The purification of tannins is a long and tedious process and to date no one has succeeded in obtaining wattle tannin entirely free from extraneous substances.

Wattle was purified in various ways, an effort being made to obtain a sample having as high purity as possible under the limitations of time and equipment in yield sufficient to provide material for forming derivatives which could in turn be purified.

A sample at one stage was investigated by paper ionophoresis. Under proper conditions, a small round spot was obtained having a mobility at pH =10 of 17.4 x 10^{-5} cm^2 per volt-sec. If the sample at this stage was a single substance, this was its mobility. If it was complex, then the mobilities of the constituents were all extremely close, probably indicating isomeric or very similar structures for the constituents.

It is not a simple matter to obtain certain of the tannins completely free of ash and of acetyl impurity. This was accomplished with wattle and a sample of another stage of purification gave the following analysis:

C	H	ASH	Ac	C-CH_3	MeO	Cl
61.61	4.75	None	None	None	0.44	0.18
61.34	4.75	None	None	None	0.56	0.30

The above satisfies the following formula:

$$C_{15}H_{26}O_{12} - 0.5H_2O$$

The presence of varying amounts of water in most tannins can be shown by the Karl Fisher Method. In the case of quebracho, the method corroborated a tentative empirical formula containing water. In one case, quebracho was obtained water-free.

The possible presence of a half mole of water or of one and one-half moles, as another formulation seemed to indicate, may possibly signify that the substance exists in a dimeric form with one mole of water held between two moles of tannin. Further reference to dimerization will be made under Molecular Weight, but for the present, since no absolutely pure sample has yet been prepared, the role of water is speculative. However, its presence in most tannin samples may be verified by the Karl Fisher Method.

Chromatography has been much employed in the purification of vegetable tannins. One difficulty is the problem of obtaining sufficient material for the preparation of derivatives. Another factor which is often overlooked is the fact that many pure compounds have been shown to give more than one spot when actually the substance is a single individual. This is due to the ionized and un-ionized molecules having different Rf values and both being present. Where a substance may form several ions it may show several spots.

Molecular Weight

The problem of obtaining accurate molecular weights of vegetable tannins is a different one. One can easily demonstrate that the purified tannins pass through cellophane. This speaks strongly against their being high polymers. Raffinose (M.W.=594) dialyzed through the same membrane. Quebracho has been shown to increase in molecular weight on standing in acetone. This seemed to indicate dimerization.

The wattle sample, of which the analysis was detailed previously, was acetylated and the product gave:

M. W. (Rast) 1083
M. W. (Calc.) 1041

This points to the tannins as being substances of modest molecular weight. It does not preclude dimerization, etc. in solvents. Molecular Weights of methylated and acetylated quebracho have been obtained by both Rast and solvent methods and agree with expected values. Several determinations

of the methylated derivatives of quebracho gave an average value of 678, again indicating a molecule of modest size. What does this mean if the samples actually consist of more than one individual?

Reference has been made to the ionophoretic experiments which indicate uniformity of mobility and to the dialytic experiments pointing to modest size. It seems reasonable that if there is more than one individual present their molecular weights will hover in the range found.

Molar Refraction

The molar refraction of wattle was measured on the sample designated as having one and one-half moles of water present. Using a molecular weight of 605.5, the density was found to be

$$d\,\frac{25.5}{15} = 1.465 \text{ giving an apparent volume } \left(\frac{M}{d}\right) \text{ of } 413 \text{ cc.}$$

By measuring refractive indices, using the sodium D line, the molar refraction of wattle was found to be 139 cc. By assuming a flavpinacol structure, the molar refraction was calculated from refraction equivalents as 146 cc., thus indicating reasonable agreement. This lends support to the structure assumed. It does not preclude the presence of isomers, but does argue against vastly different structures.

Surface Tension

The surface tension of wattle was measured at different concentrations and temperatures. The values obtained showed that it was not strongly surface active and that evidently no strongly surface active component was present in any significant concentration.

Viscosity

The viscosity of aqueous wattle solutions was measured at different concentrations and different rates of shear. This viscosity increased with shear. This indicated the presence of a dilatant effect or a lining up of molecules during shearing. Hydrogen bonding of a sheet-like molecule might be expected to enhance this effect.

Molecular Volume

The molecular volume of quebracho was computed from the apparent molar volume and compared with the volume obtained by measurements made on a model using the assumed structure. Since the apparent molar volume of wattle was close to that of quebracho, the computations apply

equally well to wattle. As very good agreement was obtained, this lends further support to the flavpinacol type of structure. The possible presence of isomers of this structure would make no significant difference.

Again speaking of quebracho (which applies equally well to wattle), the volume of a cylinder of rotation, having as altitude the length of the molecule in its longest planar form and as diameter the average width of the plane, is 1350A^3. Alternatively, the model may be folded and the catechol planes brought parallel giving a box-like shape. The total volume of this figure is 1320A^3. The additional space in each of these corresponds very closely to one more molecule of the compound, and, in the case of the planar model, the total volume equals that of two molecules placed adjacent plane to plane. The planar form would be conducive to hydrogen bonding to form a dimer in cases where strong hydrogen bonding is possible as here.

Acetylation

Wattle was treated to remove all acetyl impurity. Alkaline hydrolysis was used to measure the acetyl content. The possibility that alkaline hydrolysis degrades the tannin to give acetic acid was eliminated, since otherwise a null value would not have been obtained. The analytical method for determining the acetyl content was tested by acetylating quebracho to whatever value the use of radioactive acetyl chloride might give. This was then analyzed both by alkaline hydrolysis and by measurement of the radioactivity. An excellent check was obtained.

In the acetylation procedure, it is necessary to follow the order: acetic anhydride, acetyl chloride, acetic anhydride. If the maximum acetyl value is not reached, a fourth acetylation should be made with acetic anhydride. If this fails, one should start with a fresh sample of tannin. Once acetyl chloride has been used, it should not be used again as this step may exert a deacetylating effect. Naturally this is not due to the acetyl chloride, but to the base hydrochloride present. Pyridine has only occasionally been successful, as pyridine hydrochloride appears to deacetylate just as it is known to demethylate. Dimethylaniline has been uniformly successful, quinoline acts like pyridine. Acetyl chloride should not be used in the first acetylation, as it is not possible to obtain the maximum acetyl value by such an order of procedure.

The acetic anhydride should be of 99–100% quality. The tetraethyl ammonium bromide used was slightly moist. This latter fact may not be significant. Sodium acetate should not be used.

The alkaline hydrolysis method used to obtain the acetyl values consisted of refluxing the substance with 4% sodium hydroxide in 50% methanol for 30–90 minutes, removal of the alcohol, acidification with sulfuric acid and magnesium sulfate, distillation of the acetic acid and titration of the acid.

Three independent laboratories in this country obtained the same high values for acetyl contents of the acetylated tannins. Two laboratories abroad did not obtain the high values on the same sample, although presumably they used the same technique. The increase in the acetyl content was followed by analyses performed at each step.

The infrared data on wattle, acetylated wattle and deacetylated wattle agree with the acetyl analyses and indicate the gradual disappearance or reappearance of hydroxyl and keto absorption as the case may be. Should the wattle not be a single compound, but a mixture of isomers, etc., the data are still applicable and indicate the removal of all free hydroxyl in the components present.

The acetyl derivative was deacetylated and the reduction in acetyl content, and increase in hyroxyl content, confirmed by analysis and by the infrared spectra. If we assume the original derivative to be a dodecacetyl derivative of a C_{30} formulation, then the deacetylated compound would have three acetyl groups present. Such a residue of three groups which were difficult to remove was also encountered with quebracho. It points to the possibility that three hydroxyl groups may be alicylic rather than aromatic.

Methylation

When quebracho was methylated, a heptamethyl compound was obtained which could easily be demethylated to the hexamethyl compound. This indicated that probably six aromatic hydroxyl groups and one alicylic hydroxyl group were methylated and that the latter was probably the one which easily demethylated. With wattle, a heptamethyl compound was obtained. No experiments on demethylation were performed, but by analogy it is probable that seven aromatic hydroxyl groups were methylated in this case.

Summary

1. Wattle tannin has been purified. The product has not been shown to consist of a single component.

2. The mobility of the purified tannin has been measured. This may be used as a means of identification. If the purified tannin consists of more than one component, then either the components form a complex or the individuals have practically the same mobilities. This points to similarity of structure, possibly stereoisomers or positional isomers. The flavpinacol type of structure allows of several stereoisomers. Those which are not enantiomorphs should be separable by chromatographic means.

3. The purified tannin sample was obtained completely free of ash and acetyl impurity.

4. The presence of a small percentage of water not removed by ordinary methods of drying can be shown by the Karl Fisher Method.

5. Increase in molecular weight with aging in a solvent has been demonstrated for quebracho and may be assumed probable for wattle. Molecular weight measurements in camphor and solvents on derivatives of wattle by the author agree. If the sample is complex, they represent an average value. Isomers would have the same value.

6. Passage of the tannin through cellophane puts an unknown upper limit on its molecular weight. It is probable that high polymers are not present.

7. Experimental determination of the molar refraction gave a value in reasonable agreement with that calculated from refraction equivalent based on a flavpinacol structure. Stereo or positional isomers have the same calculated value.

8. Surface tension measurements indicated the absence of any strongly surface-active component.

9. The molecular volume agreed with measurements made on the structure assumed. A possible layout of the model favoring dimerization was postulated.

10. Acetylated and methylated derivatives were prepared. Empirical and structural formulas were assumed for wattle and its derivatives. If wattle is complex, but the components are merely stereo or positional isomers, the assumptions are allowable. If the components differ in other aspects, the structures are invalid, but the degree of acetylation and methylation are significant.

11. Infrared data agree with the analytical work on wattle and its derivatives.

Received August 30, 1955.

Studies on Leather by Means of a Sonic Technique*

By Joseph R. Kanagy and Myron Robinson

Abstracts

Nondestructive studies utilizing measurement of the velocity of sound through leather have been made. It is shown that sound velocity varies substantially with changes in chemical and physical structure and particularly with fiber orientation. Accordingly, velocity measurements are indicative of modifications of the fibrous order produced by strain, aging, and filling. There is good correlation with the results of tensile and breaking elongation tests. The effects of tannage, grease, and moisture can also be demonstrated. Hide-to-hide and point-to-point variations are extensive and several simultaneous factors influencing velocity are usually not readily distinguishable.

The speed of sound through leather increases with period of aging at 100°C. until it reaches a maximum. Therefore, the proximity of a sample of leather to the maximum may

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indicate its quality. For an inhomogeneous material such as leather the sonic technique has the distinct advantage of providing a means of following the effects of aging, chemical treatments, and the like on a single specimen.

1. Introduction

In recent years the use of sonic techniques for the non-destructive determinations of certain mechanical properties of high polymers has become widespread [1*]. Such methods have utilized frequencies ranging from less than one cycle/sec. up to the lower megacycle region. A broad frequency range is necessary in order to effectively study variations in the dynamic mechanical characteristics, modulus of elasticity, and internal dissipation, which are frequency dependent.

Sonic measurements reported in the literature have involved almost exclusively sinusoidally time dependent continuous wave, or at ultrasonic frequencies, pulsed transmission [2]. Use of a single known frequency readily permits mathematical analysis of the data and its resolution into basic physical constants characteristic of the viscoelastic behavior of the material. Sonic methods, furthermore, in addition to furnishing a means for securing certain fundamental information, have been successfully utilized in nondestructive testing. This work has been chiefly concerned with the location of flaws in a wide variety of manufactured products [3].

The change in acoustical resistance offered by flaws reflects a portion of the incident sound energy, and examination of either the reflected wave [4] or the transmitted portion indicates the existence of a discontinuity in the material. A test based on this principle has recently been successfully employed to locate internal defects in tires [5]. The use of sound velocity measurements for production quality controls in the manufacture of paper, yarn, and soap, has also been reported [6]. In testing applications, it should be noted that viscosity and frequency considerations are relatively unimportant since only relative measurements are required to establish the deviation of a sample from its control.

If a longitudinal (compressional) disturbance is propagated along a medium whose transverse dimensions are small compared to the wavelengths involved, then the following well-known relation holds:

$$E = v^2\rho$$

where v is the sound velocity, ρ the density of the propagating medium, and E is Young's modulus, the latter being defined as the force per cross-sectional area required to produce a given elastic strain.

If the material shows appreciable acoustic attenuation as indicated by a substantial decrease in the amplitude of the sound wave in the course of its travel through the specimen, the simple relationship given above is no longer

*Figures indicate the literature references at the end of this paper.

suitable for an accurate determination of the modulus. Frictional losses associated with the damped sound wave may be expressed in terms of a viscosity coefficient, or coefficient of internal friction, and a correction factor based on this coefficient, the wave frequency, and the velocity, is subsequently applied to the right-hand side of the equation [2], [7]. For perfectly elastic media, such as metals, the correction is unity, for nylon at 10 kilocycles/sec., it is about 0.95 [8], and for carbon black buna-N rubber under a tension of 800 g/cm.[2], about 0.14 [2].

Because of the complex nature of the transmitted wave only uncertain frequency and attenuation measurements are possible with the present technique. Consequently, the correction factor for leather cannot be effectively determined and it is not feasible to express velocities in terms of moduli. This leather correction factor, however, is significantly less than unity. It is clear that in testing applications of the present kind attenuation and frequency considerations are of secondary importance, since only relative velocity measurements are required to establish the deviation of a sample from its control. Data in this report, therefore, are presented in terms of the experimentally more directly measured quantity, velocity.

The application of sonic testing techniques to leather at the National Bureau of Standrads was undertaken at the request of the Office of the Quartermaster General and had as its goal the evaluation of sonic methods for the nondestructive testing of leather. Sound velocity was studied as a function of several variable properties associated with leather, namely, location

FIGURE 1.—Essential components of the Pulse Propagation Meter.

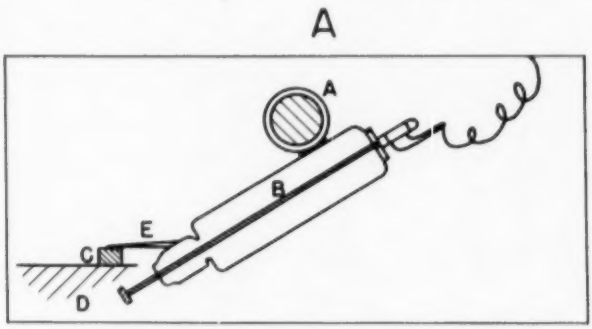
and direction in the hide, tannage, strain, aging, grease and moisture content, fiber orientation, tensile strength, and ultimate elongation.

2. Method of Procedure

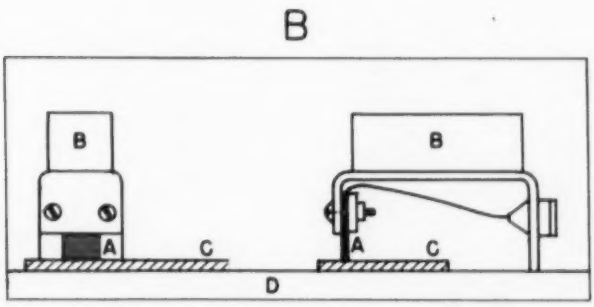
2.1 *Apparatus*

The Pulse Propagation Meter, or PPM [9], is an instrument designed to measure the time of transmission of a damped wave train through a sound propagating medium. The essential components of the apparatus are shown by the simplified block diagram, figure 1.

The output of a 100,000-cycle/sec. crystal oscillator is passed through a series of frequency dividers reducing the frequency to 160 pulses/sec. These



A, BEARING; B, PHONO PICKUP CARTRIDGE
C, SPECIMEN; D, CORK BED; E, STYLUS



A, CRYSTAL; B, WEIGHT; C, SPECIMEN
D, CORK BED

FIGURE 2.—Methods used to mount crystals on specimens.

pulses are then intensified in the pulse generator for application to the transmitting crystal (transducer). Each electrical pulse shocks the piezoelectric crystal into oscillation at frequencies determined by the nature of the pulse itself, the crystal, its restraining mounts, and the specimen. The wave train produced by crystal excitation is transmitted through the specimen, received by the crystal pickup at the opposite end, and applied to an amplifier. The amplified signal is next fed to a timing circuit which had previously been triggered by the outgoing pulse and which now delivers an output proportional to the time interval between its two inputs, namely, the time required for transmission through the specimen. The propagation time in microseconds may be read directly on a meter or automatically recorded.

Two types of transducers have been used in applying PPM signals to leather specimens under test. Either direct contact by means of a bare piezoelectric crystal or indirect contact through a commercial phonograph pickup needle may be used to advantage, figure 2. The latter possesses the ruggedness of construction necessary to insure long life and is particularly adapted to testing yarnlike specimens of small cross section. Unfortunately, mechanical coupling between the crystal and the needle, delay the sound signal both in transmission and reception, and a time delay of as much as 100 microsec. may be incorporated into the meter reading.

Since the PPM can be read on either a 100- or 500-microsec. maximum scale, a 100-microsec. time delay renders the smaller scale valueless. To overcome this difficulty and reduce delays to a minimum, mechanical connecting links between the electrical circuit and the specimen should be dimensioned to have as high a resonant frequency as possible[10]. In practice, Rochelle salt twisters and barium titanate benders (bare except for suitable holders and a moisture protective jacket over the Rochelle salt crystals) were applied directly to the leather specimen, but these bimorphs (two individual crystal plates cemented together to give a characteristic type of vibration, e. g., bending or twisting) were rejected for the more satisfactory zero degree, cross-cut, Rochelle salt, single plate crystals. These, because they vibrate in a direction parallel to their own plane, are suitable for leather strips; whereas, bimorphs vibrating perpendicular to their broad plane would prove more desirable for yarnlike materials. The crystal mounting is shown in figure 2. Leads must be carefully shielded to prevent radiation signals from triggering the timing circuit, especially when the crystal mounts are close together.

2.2 *Measurement of Velocity*

The meter reading is a composite indication of both the time required for a sonic disturbance to be propagated between transducers and the various electrical and mechanical time delays between the specimen and the timing circuit. The electrical delays for the instrument are constant and small. On

the other hand, the mechanical delay encountered in the bare piezoelectrical crystal or crystal phonograph cartridge is dependent on the transducer's own characteristics, the pressure with which it rests on the specimen, and the specimen's geometry and structure. Any factor that can be expected to affect the loading of the transducer will vary the magnitude of the initial time delay. Because of the variety of circumstances influencing it, the initial delay, if required, must be determined individually for each specimen. Velocity is determined by measuring the reciprocal slope of the propagation time versus distance curve.

Some curves obtained for leather are shown in figure 3. Situation (A) is usual, although occasionally the discontinuity shown in (B) occurs. This is due to the attenuation of the wave with distance and the correspondingly modified wave train which no longer triggers the PPM timing circuit with the same oscillation that it did previously. The slope remains unchanged.

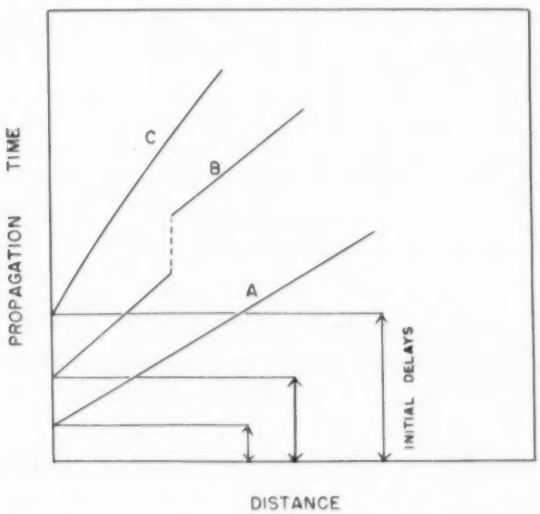


FIGURE 3.—Several types of propagation time versus distance curves obtained from leather.

Points on the curve were either determined individually and plotted manually or one crystal transducer was moved at a constant speed along the specimen while the other transducer remained fixed, and an automatic recording was made. The automatic method facilities rapid measurement, but vibrations set up by moving parts had to be eliminated to prevent erratic behavior by the PPM. This was not always possible and manual operation was occasionally unavoidable. For any single velocity determination, the

initial delay must be kept constant if the points for a homogeneous specimen are to fall on a straight line. For this reason, transducer pressure against the specimen must not be altered in the course of a velocity determination. This is simply done by allowing the transducers to rest under their own weight against the specimens. When phonograph pickups are used, the length of needle in contact with the specimen must remain unchanged if the length of the sound path through the needle, and hence the initial delay, is to remain fixed. Sometimes the curve of figure 3(C) is found to result. Whether this is a consequence of variable delay or variable velocity cannot be readily ascertained.

The apparatus employed to give a continuous recording of propagation time vs. transducer separation is shown in figure 4. The device is essentially a modified lathe bed containing one transducer mounted on the tail stock and the other on the carriage. The leather specimens may be kept under constant tension at a given strain while one transducer remains fixed and the other is moved to make contact with the specimen anywhere along its length.

Before measurements were made, all specimens were conditioned to equilibrium at 73° F. and 50 per cent R. H.

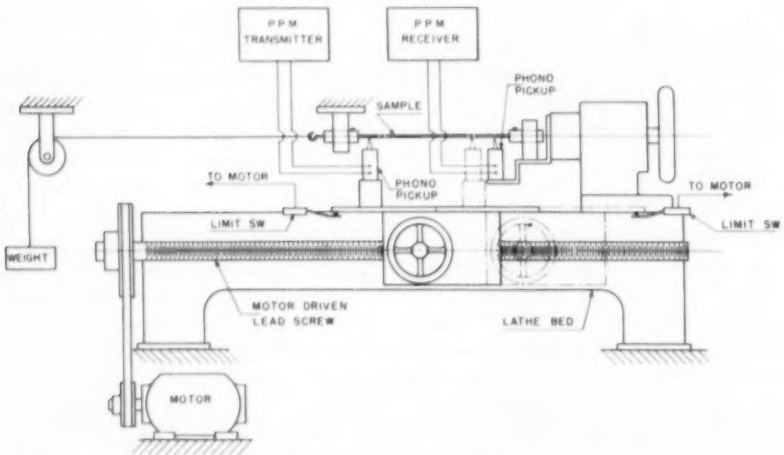


FIGURE 4.—Apparatus employed to give a continuous recording of propagation time versus transducer separation.

3. Experimental Results

3.1 *Study of Wave Shapes*

An oscillogram of a typical PPM wave train in upper leather is shown in figure 5. The time duration of the wave train is a maximum of 6250 micro-

seconds. The frequency spectrum varies somewhat with the specimen, but in general the principal component is in the range of 3000-4000 cycles/sec.

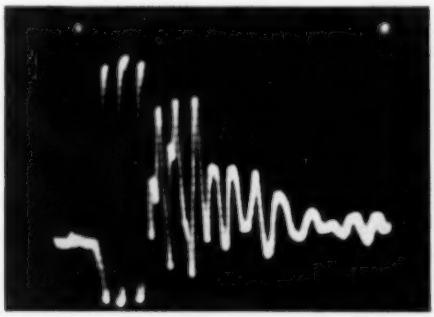


FIGURE 5.—Oscillogram of a typical PPM wave train in leather. The first 3700 microseconds only are shown.

It is essential that almost complete damping of any particular wave train occurs in the 6250 microseconds provided between pulses in order that the timing circuit be able to distinguish between adjacent trains. Metals, because of their low damping rates, are prone to ring excessively and introduce serious difficulties into PPM tests.

3.2 *Attenuation Constant*

Since the PPM timing circuit measures the transmission time of each wave train individually after the previous wave has dissipated itself, there is no reflection of preceding waves with which to be concerned. The signal amplitude may then be assumed to diminish along the transmission line specimen so that its magnitude at distance d from point d_o is $A_o e^{-\alpha d}$, where A_o is the amplitude at d_o and α is the attenuation constant. α is commonly measured in nepers/cm., one neper being a reduction in amplitude by the factor 1/e. α is 1/8.686 times the uniform attenuation in decibels and is readily shown to be equivalent to 2.303 $\log_{10}$ (amplitude at d cm./amplitude at d + 1 cm.).

Measurements of attenuation, by observing on the oscilloscope the decreasing amplitude of a particular section of a wave train as the transmitter and receiver crystals are separated, were not highly satisfactory; both the amplitude and the frequency of the waves lacked the required uniformity. However, on the basis of rough measurements, the attenuation constant, α, for upper leather is estimated at 0.1–0.2 nepers/cm. by this method.

It is interesting to compare the order of magnitude of the attenuation constants obtained for leather with those for other polymeric materials at 3000 cylces/sec. and room temperature: polyethylene, 0.012; nylon (undrawn

filament), 0.003; Neoprene GN at 6 per cent strain, 0.51. These results indicate that the acoustic attenuation characteristics of leather lie midway between that of fibers and rubberlike materials. A likeness between rubber and leather exists in that the velocity of sound through both materials is significantly lower than through polythylene or nylon. These conditions are probably indicative of a degree of correspondence in internal structure between leather and the rubbers not to be found between leather and the other more crystalline materials.

3.3 *Influence of Specimen Geometry*

Experimentally, the velocity of sound transmission is found to vary with the geometry of the propagating specimen. Effects of the specific parameters investigated, thickness and width, are shown for retan upper leather in figure 6. Parallel displacement of the curves is justified by the failure of a width

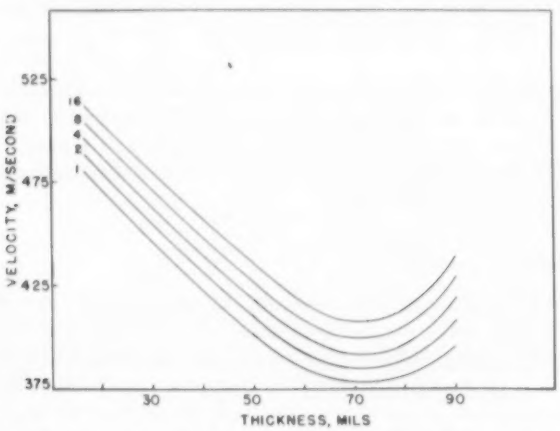


FIGURE 6.—Velocity as a function of thickness and width. Width in cm. is indicated by the numbers in the upper left corner. Curves indicate average results from single specimens from nine different hides.
USXL* 113: 3 per cent sulfur dioxide reduced chrome; 40 per cent retannage; 25 per cent grease. Location "O".** Direction of propagation: ⊥backbone.

*United States Army experimental leather.
**The "O" section is just forward of the mid-section of a side. See figure 23.

thickness interaction to manifest itself in an analysis of the data. Wide fluctuations in velocity from hide to hide in the nine hides examined at each width tended to obscure the functional relationships; hence the experimental points are not indicated. The data do give some indication that the 2-cm. curve may lie above the 4-cm. curve, but the evidence is not conclusive and the anomaly is not shown in the figure. Different velocity minima are ob-

served for two vegetable leathers in figure 7. A number of upper leathers studied showed no increase in velocity at widths above 16 cm. (figure 8). Because of this it seemed desirable to cut specimens to a minimum 16-cm. width, but this frequently proved impractical experimentally.

From a practical test viewpoint, these findings are highly significant. It is shown to be absolutely necessary that geometric uniformity in specimens be maintained if effective comparisons are to be made.

The reasons for dimensionally-caused velocity variations are not definitely known. Several possibilities present themselves:

(1) If the effective frequency of the transmitted waves is taken to be 3000–4000 cycles/sec. and the commonly encountered velocities in unstrained specimens 200–500 m./sec., then the associated wave lengths will be about 5–17 cm. This roughly coincides with the velocity-sensitive range of widths. Suppose the lateral dimensions of the propagating medium which are initially small compared to the wave length subsequently increase in size. The wave no longer will be transmitted only in one direction, the mechanics of propagation will be modified, and the velocity will consequently change. Although the direction of velocity change with changing width is in accord with theory, the velocity variation with thickness remains unaccounted for. If sound propagation were entirely a bulk leather phenomenon, the velocity would be expected to vary directly with thickness since stiffness increases with the thickness.

(2) One might expect, and much of the evidence does indeed indicate, that sound propagation in leather is in effect a fiber rather than a bulk leather phenomenon. For example, Young's modulus E, calculated from the classical relationship $E = v^2 \rho$, where v and ρ are sound velocity and fiber density, respectively, is of the order of magnitude of 10^9 dynes/cm^2. This is in agreement with static modulus values for fibers [11], and both these values are of a higher order of magnitude than Young's modulus for bulk leather.

It is, however, most probable that the fibers behave in an indirect fashion to influence the passage of sound through leather. The wave length of the transmitted pulse is much greater than the distance between fibers in the bulk leather. On the other hand, the bulk leather of cattlehide contains many air pockets which tend to soften the leather and decrease its modulus. This would tend to decrease the speed of sound by setting up vibrations in different directions. If the fibers are oriented in the direction of propagation, the modulus of the bulk leather would be higher and it is expected that the speed of sound in that direction would likewise be greater and would be less affected by the width of the specimen.

To examine the above hypothesis, tests were run on vegetable-tanned sharkskin. Sharkskin fibers occur naturally in a highly oriented basket-type weave, the two fiber axes being mutually perpendicular in the plane of the hide.

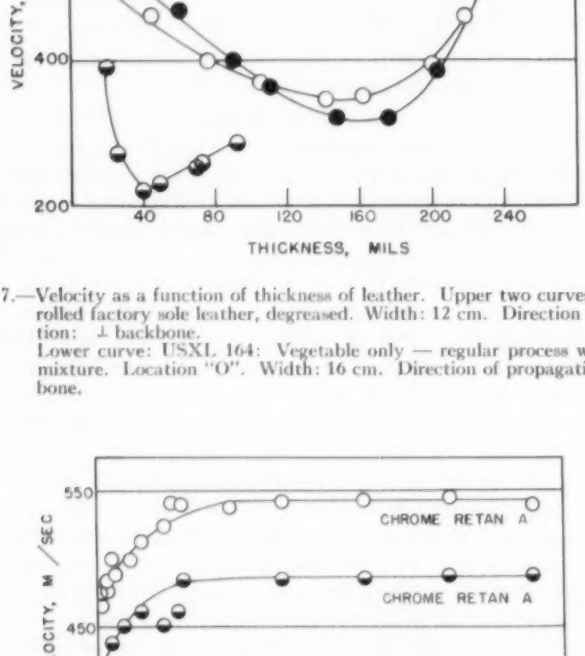


FIGURE 7.—Velocity as a function of thickness of leather. Upper two curves: Finished rolled factory sole leather, degreased. Width: 12 cm. Direction of propagation: ⊥ backbone.
Lower curve: USXL 164: Vegetable only — regular process with stuffing mixture. Location "O". Width: 16 cm. Direction of propagation: // backbone.

FIGURE 8.—Velocity as a function of width for three upper leathers. The upper two curves are from different hides of the same tannage.
Upper two curves: USXL 212: Chrome-retanned with Chemtan. Location "O". Thickness: 95 mils. Direction of propagation: ⊥ backbone. Third curve down: USXL 130: 3 per cent glucose reduced chrome and formate; 20 per cent retannage; 20 per cent grease. Location "O". Thickness: 95 mils. Direction of propagation: ⊥ backbone.

Specimens were cut so that sound transmission could be effected in the direction of a fiber axis and at a 45° angle with the axis. The specimens cut at an angle of 45° with the fiber axis have in accordance with this hypothesis sound paths roughly comparable with those existing in cattlehide, and a similar velocity-width relationship is expected. On the other hand, specimens with the fibers oriented in the direction of sound propagation should show no velocity decrease upon decreasing the width of the specimen. Figures 9 and 10 give the experimental data for the cuts of sharkskin described. The data of figures 9 showing the 45° cut behave as expected; figure 10 shows that within experimental error the velocities parallel to the fiber axis are independent of width. The velocity of parallel propagation is also observed to be greater than the maximum oblique velocity.

(3) The velocity-thickness variation is still unexplained and may be due, at least in part, to the changing composition of the propagating medium as flesh layers are split off.

3.4 *Effect of Strain*

Velocity of sound propagation as a function of the strain was studied for a number of upper leathers. The lathe bed previously described was used in these tests. The results on specimens about 80 mils thick and 60 mils wide are given in figures 11 and 12. The aged specimens were degreased in chloroform prior to accelerated aging at 100°C. for 7 days.

The velocity-strain dependence of leather is similar to that found in other long chain polymers. In such materials the mechanical behavior is considerably altered when molecular chains become oriented under stress. In general, the elastic modulus, and therefore sound velocity, is found to be much greater for highly oriented materials than for such materials in the isotropic state[7, 12]. Low velocity values in the unstrained condition are ascribed to randomly oriented or kinked chains or fibers. After initial orientation, chrome-retanned leather is less prone to further orientation under stress and the velocity-strain curves level off as shown. At breaking strains of 30–50 per cent, velocity increases of 40–400 per cent over the unstrained values have been observed. In view of this behavior it does not appear probable that the modification of internal structure resulting from strain is entirely a fiber phenomenon. The orientation probably occurs on both the fibrous and subfibrous, perhaps molecular levels.

The fibers of kangaroo tail tendon occur naturally in a highly oriented state and it may, therefore, be expected that the velocity of sound in the tendon will be comparatively high. Measurements show this velocity to be about 2000 m./sec. and unaffected, within the experimental error, by tensions of as much as 500 kg./cm.^{2}, which cause no appreciable strain in the kangaroo tail. The comparison of this behavior with that of hide discussed in

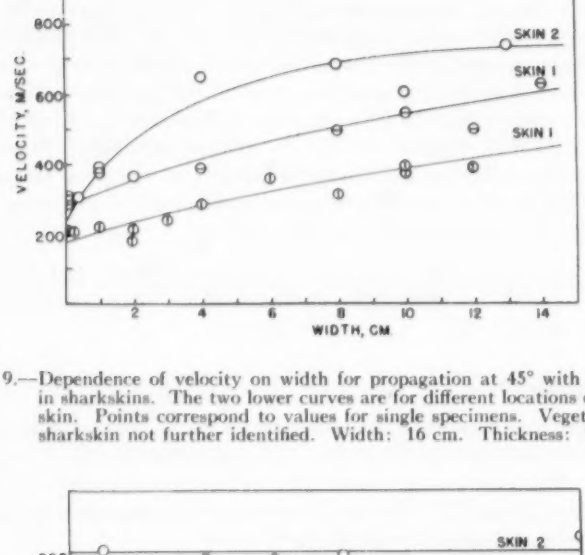


FIGURE 9.—Dependence of velocity on width for propagation at 45° with a fiber axis in sharkskins. The two lower curves are for different locations on the same skin. Points correspond to values for single specimens. Vegetable-tanned sharkskin not further identified. Width: 16 cm. Thickness: 130 mils.

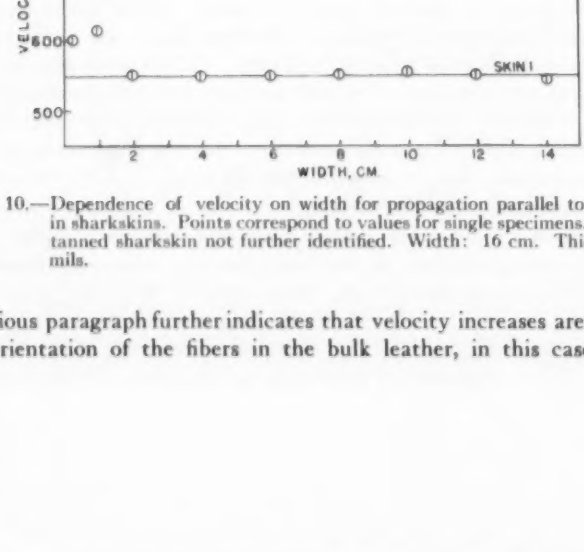


FIGURE 10.—Dependence of velocity on width for propagation parallel to a fiber axis in sharkskins. Points correspond to values for single specimens. Vegetable-tanned sharkskin not further identified. Width: 16 cm. Thickness: 130 mils.

the previous paragraph further indicates that velocity increases are attributable to orientation of the fibers in the bulk leather, in this case, through strain.

Velocity measurements made during the stretching and subsequent recovery of leather exhibit a permanent change in structure due to strain. This condition is illustrated in figure 13 for a retanned upper leather specimen. At elongations of 20 and 30 per cent the stress was removed from the specimen and essentially complete relaxation permitted to occur. Although the stretched leather displays the tendency to return to some prior dimensional status, the relaxation and stretching paths do not coincide. Presumably the fibers oriented in the stretching process are kept in alignment by the applied stress, thereby yielding comparatively high velocities, but release of the stress allows the permanently rearranged condition of the fiber matrix to manifest itself in lower velocities, probably by a decrease in the modulus.

In the course of straining upper leather, velocities rise at constantly decreasing rates (with some apparent exception at small strains), but the single all-vegetable-tanned leather tested shows this behavior not to be universal.

The more shallow initial climb of the vegetable velocity-strain curve, figure 14, is indicative of the greater resistance of that leather to orientation under stress. The relatively dense packing of interfiber regions with vegetable tanning materials may account for this behavior. It is known, in this connection, that the presence of water solubles is manifested in reduced velocities. Four vegetable crust specimens were soaked in water for 70 hours, thereby removing about 8 per cent in solubles of the original total weight. The high velocity increases resulting are listed in table 1. It is probable that the fibers have returned to a more oriented condition or that the water-soluble materials act in some way to decrease the velocity.

TABLE I

Effect of Water Solubles on Vegetable Crust Leather*

Specimen	Velocity		Water solubles lost; per cent of initial total weight
	Initial	After soaking	
1. //	288	487	8.7
⊥	241	440	
2. //	320	381	6.5
⊥	290	476	
3. //	417	695	7.8
⊥	351	588	
4. //	357	635	7.8
⊥	290	580	

*Vegetable crust leather: Width: 16 cm. Respective thicknesses: 53, 75, 40, and 36 mils. Four adjacent specimens cut from center backbone region.

The slightly slower initial velocity increases with strain, just barely incated by the plotted points in figures 11, 12, and 13, may be real although they were ignored in drawing the curves. In such cases it is possible that for

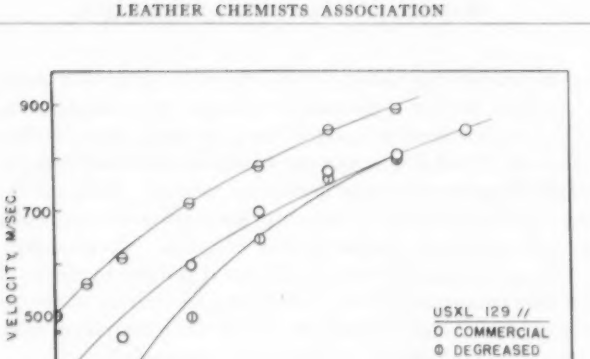


FIGURE 11.—Variation of velocity with strain for chrome-retanned leather, parallel direction. Each point is an average of at least six specimens through 20 per cent elongation for aged and commercial samples and through 25 per cent elongation for the degreased sample. Other points are averages of less than six specimens.
USXL 129: 6.5 per cent sulfur dioxide and formate ; 60 per cent retannages; 30 per cent grease originally. Location "O". Width: 60 mils. Thickness: 80 mils.

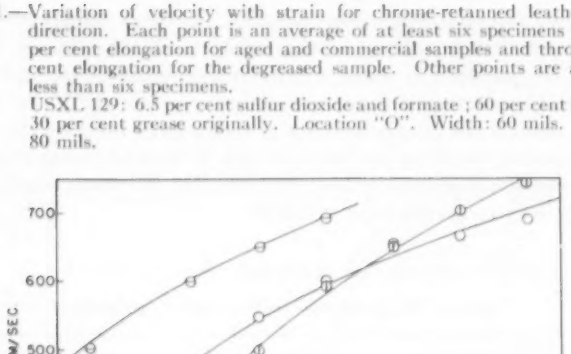


FIGURE 12.—Variation of velocity with strain for chrome-retanned leather, perpendicular direction. Each point is an average of at least six specimens through 15 per cent elongation for the aged sample and through 25 per cent elongation for the commercial and degreased samples. Other points are averages of less than six specimens.
USXL 129: 6.5 per cent sulfur dioxide and formate chrome; 60 per cent retannage; 30 per cent grease originally. Location "O". Width: 60 mils. Thickness: 80 mils.

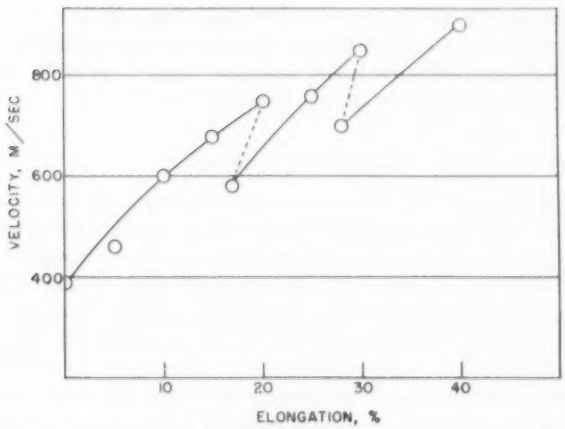


FIGURE 13.—Variation of velocity with elongation for chrome-retanned upper leather during alternate straining and relaxation.
Chrome-retanned upper leather not further identified. Width: 60 mils. Thickness: 80 mils.

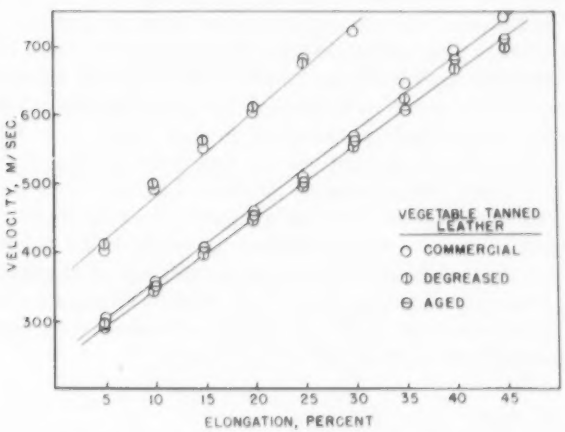


FIGURE 14.—Variation of velocity with strain for a vegetable leather of unknown history. Upper set of points, parallel direction. Lower set of points, perpendicular direction. This is the only leather tested which shows no effects of aging on velocity. Each point is an average of at least six specimens through 15 and 30 per cent elongation, respectively, for degreased and commercial in the parallel direction, and through 35 per cent elongation for commercial and degreased and through 45 per cent elongation for aged in the perpendicular direction. Other points are averages of less than six specimens. Vegetable leather not further identified. Original grease content is unknown. Width: 60 mils. Thickness: 45 mils.

moderate strains the fibers or molecular chains may be able to move more freely than in the unstrained material. Also, it has been observed with other materials that crystalline or other oriented regions existing initially are broken up by the application of low stresses[13].

3.5 *Correlation of Tensile Strength with Velocity.*

The tensile strength of a hide varies considerably with orientation of the test specimen with respect to the backbone from location to location. Belly fibers, for example, are known to be highly oriented in the parallel direction, and on that account to possess a high tensile strength in that direction. In the region of the tail the orientation and greater tensile strength are principally in the perpendicular direction[14]. It may be supposed, therefore, that sound velocity which is highly dependent on fiber orientation will furnish a suitable nondestructive measure of tensile strength. To determine whether or not this is so, velocity and tensile strength measurements were made on two belly and two butt specimens cut from each of three chrome-retanned sides 85 ± 15 mils thick. The resulting data are graphed in figure 15.

The coefficient of correlation between the velocity and the tensile strength is 0.84. It must be noted, however, that sonic measurements may be used as a basis of comparing tensile strengths only in specimens of the same tannage, geometry, and history. The higher velocities found in aged leathers, for example, may, where tensile strength considerations are involved, be compared only with similarly aged leathers and not with untreated varieties.

It has been shown that tensile strength and breaking elongation for a particular leather are inversely proportional to each other[15]. It follows, then, from previous considerations that velocity and breaking elongation must also be inversely related. Measurements do show that, in accord with this, the velocity-breaking elongation curve has a negative slope (figure 16). Physically this is interpreted to mean that a relatively unoriented (low velocity) leather will not break on stretching until after some measure of orientation has occurred. Conversely, a highly oriented (high velocity) leather cannot undergo extensive reorganization of its fiber matrix to accommodate an applied stress, and consequently must rupture under lower strain.

3.6 *Effect of Aging*

To determine the usefulness of the PPM in the nondestructive measurement of deterioration, a number of upper leather specimens 0.085 inch by 2 inches by 6 inches were aged at 100°C. for a total of 294 hours. Velocity measurements were made at convenient intervals and prior to each measurement the specimens were allowed to return to standard conditions of temperature and humidity. The data are plotted in figures 17 and 18. Velocities increase with duration of time of aging, grease content, and parallelism with

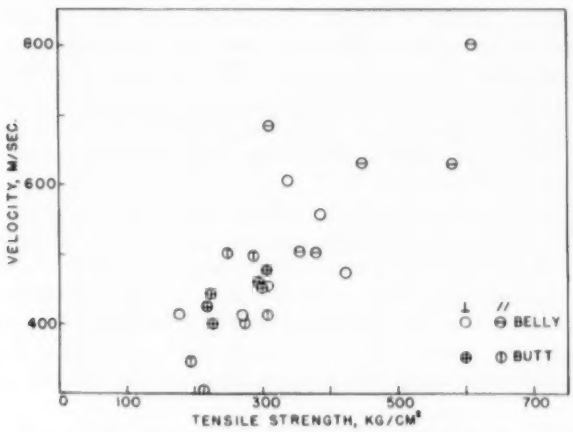


FIGURE 15.—Correlation between velocity and tensile strength for chrome-retanned upper leather.
Chrome-retanned upper leather not further identified. Specimens cut in dumbell shape ½ inch wide at neck and 1 inch wide at ends. Thickness: 80 mils.

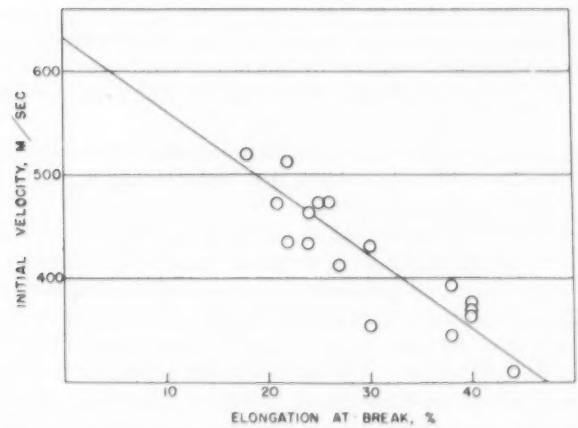


FIGURE 16.—Correlation between initial velocity and ultimate elongation for chrome-retanned upper leather. Same side and locations as in figure 15. Width: 0.35 cm. Thickness: 80 mils. Sound propagation and stressing are // backbone.

the backbone. The latter behavior is due to the fact that the specimens were taken from just behind the mid-shoulder region where fiber orientation parallel to the backbone predominates over perpendicular orientation. In

the region of the tail the reverse is true and velocities are conversely affected. Early stages of aging have the most effect, the velocities seemingly approaching maxima beyond 200 hours. The increase in velocity may be attributed either to a shortening of the sound path by a contraction of fibers (a velocity rise is observed in shrunken leathers), or on the microscopic scale, to a modulus increase resulting from changing configuration and structure of the chain molecules.

It is of particular importance to consider the suitability of the PPM as a means of effectively distinguishing between deteriorated and undeteriorated leathers. Probably the best criterion would be to determine whether the velocity had reached a maximum or how near it was to a maximum. Even if it can be assumed that the substantial between-hide variations interpose no complications, the complete history of a specimen would be required before extent of degradation could be estimated accurately in terms of velocity.

3.7 *Directional and Locational Variability*

It is evident that sound velocity measurements in leather reveal the same high degree of variability exhibited in general by physical tests. Substantial differences are shown to exist between similar hides, between different locations in the same hide, and between different directions of sound transmission in a single location. Figure 19 illustrates the velocity fluctuations measured with the crystals separated 8 cm. in the direction perpendicular to the backbone down the middle of each of three bends of sole leather. The marks on the bend in the figure indicate the points at which measurements were made.

Although individual differences are sizable, the over-all velocity trend is not obscured. The pattern is roughly that which would be predicted on the basis of destructive physical testing.

Sound velocity as a function of direction of propagation through a total angle of 180° with the backbone is shown in figure 20. Velocities were determined for one butt and one belly location on the same chrome-retanned hide. As already pointed out, perpendicular orientation of the fibers is dominant in the region of the butt; whereas, parallel orientation with the backbone is general in the belly region. The indicated oval-shaped curves showing the dependence of velocity on direction are, therefore, to be expected. The curves are not altogether smooth despite the fact that measurements were made by rotating the crystals over an area of only 10 cm diameter. The smaller irregularities may be due to uncertainties of measurement, but the larger ones undoubtedly reflect real differences in structure even in areas close together.

3.8 *Effect of Moisture.*

In view of the fact that velocity in leather is so markedly influenced by fiber direction, it is interesting to note that no corresponding effect has been

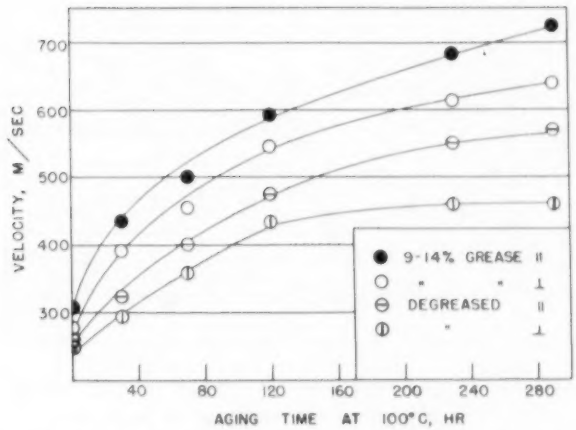


FIGURE 17.—Velocity as a function of accelerated aging at 100°C. for chrome upper leather.
USXL 156: 4.5 per cent sulfur dioxide and formate ; 9-14 per cent fatliquor originally. Location "O". Width: 5 cm. Thickness: 85 mils.

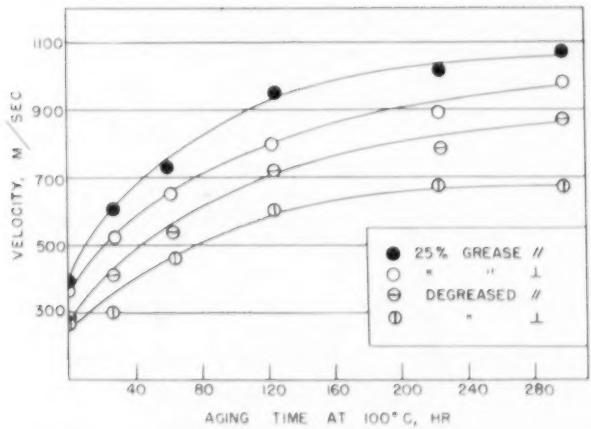


FIGURE 18.—Velocity as a function of accelerated aging for chrome-retanned upper leather. Single specimens used for each curve.
USXL 110: 6.5 per cent glucose reduced chrome; 40 per cent retannage; 25 per cent grease originally. Location "O". Width: 5 cm. Thickness: 85 mils.

observed with fresh beef muscle[16]. It is found that velocity values obtained with an ultrasonic beam traversing the tissue perpendicular to the long axis of the muscle bundles do not differ appreciably from those measured with the

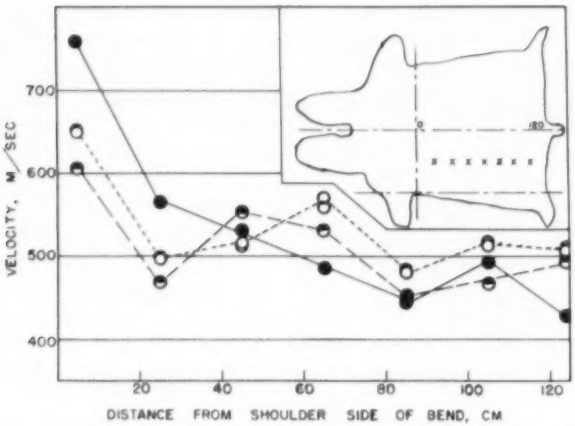


FIGURE 19.—Dependence of velocity on location in three bends of sole leather. Crosses in the hide diagram indicate points at which velocities are measured. Points shown as *O* are duplicated readings.
Finished rolled factory sole leather. Thickness: 210-260 mils. Measurements made in situ on whole bends.

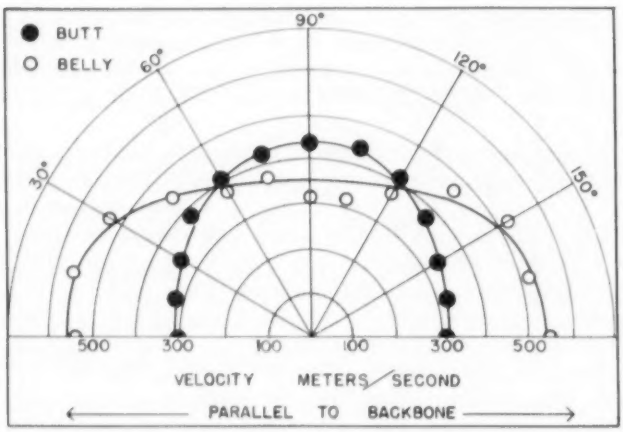


FIGURE 20.—Dependence of velocity on direction of propagation with respect to the backbone in chrome-retanned upper leather.
USXL 113: Thickness: 85 mils. Measurements made in situ on whole side.

energy directed parallel to the muscle fibers. This phenomenon is to be accounted for in terms of the very high water concentration in fresh meat since the measured velocity through the beef is approximately the same as

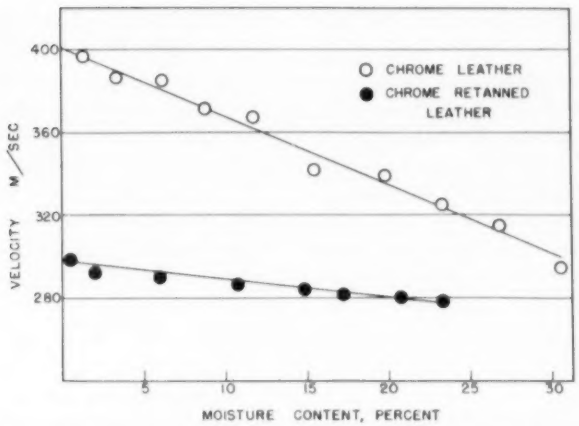


FIGURE 21.—Velocity as a function of moisture content in chrome and chrome-retanned upper leather.
Lower curve: USXL 113, degreased. Location "O". Width: 16 cm. Thickness: 75 mils.
Upper curve: USXL 156, degreased: 4.5 per cent sulfur dioxide and formate. Location "O". Width: 16 cm. Thickness: 75 mils.

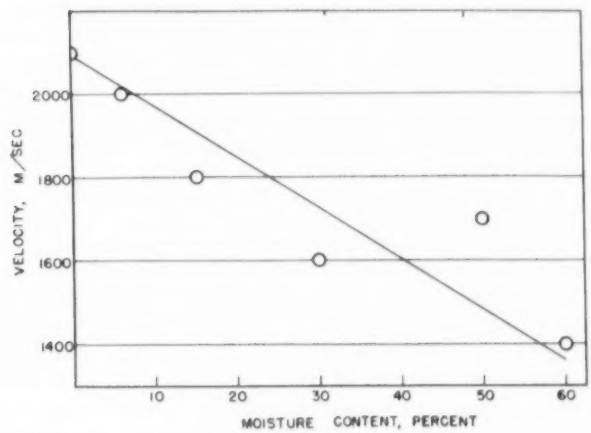


FIGURE 22.—Velocity as a function of moisture content in kangaroo tail tendon.
Kangaroo tail tendon, rawhide, about 0.01 cm.^{2} cross section.

through water. Corresponding results might be expected with moist leather, but experimentally this is not seen to be the case. Figure 21 illustrates the behavior of chrome and chrome-retanned upper leathers, both of which were

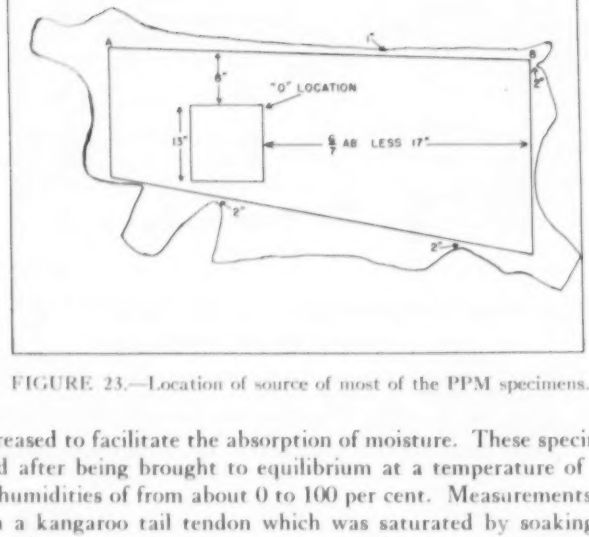


FIGURE 23.—Location of source of most of the PPM specimens.

first degreased to facilitate the absorption of moisture. These specimens were measured after being brought to equilibrium at a temperature of 73°F. and relative humidities of from about 0 to 100 per cent. Measurements were also made on a kangaroo tail tendon which was saturated by soaking in water and which was subsequently dried to equilibrium at standard conditions (figure 22). In order to keep the tendon taut during the drying process, it was necessary to keep it under a tension of 700 g. The scatter of the data is a result of the cross-sectional and surface irregularities of the specimen. As with the leather, velocity is a descending function of moisture content.

4. Summary and Conclusions

Velocity of sound propagation through leather is observed to vary substantially with changes in chemical and physical structure and particularly with fiber orientation. For this reason, sound velocity measurements may be used to follow the various physical modifications, strain, shrinkage, aging, etc., affecting the fibrous order. For an inhomogeneous material such as leather this has the distinct advantage of providing a means of following the effects of aging, chemical treatments, and the like on a single specimen. The appreciable between-hide and position-on-hide variation occurring in all other physical tests of so inhomogeneous a material as leather is duplicated in sonic studies, and severely restricts the utility of individual measurements. Nevertheless, under certain limited conditions, nondestructive velocity tests show good correlation with destructive tensile strength or breaking elongation tests inasmuch as the latter two are also functions of orientation. The effects of tannage, grease, and moisture can also be effectively demon-

strated; however, several simultaneous effects are usually not individually distinguishable by sonic examination.

Although sonic studies show only limited promise in investigations of a practical nature involving leather, the use of known frequencies under controlled laboratory conditions may be extremely valuable. Knowledge of the dynamic mechanical properties of a material over a range of environmental conditions is essential to characterizing its structure. Sonic studies with leather are capable of contributing to our present understanding of the mechanical behavior and deterioration of collagen and other long chain molecules.

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ABSTRACTS

Curtailing Tan Waste. *Lea. Trades Rev.*, **97**, 65 (1955). Loss of tans during processing is of obvious importance. In the past a 30 percent loss was not unusual. Today a loss of over 20 percent is considered bad. A comparison of present day losses with those of 50 years ago follows;

	Modern	Old
(1) Loss due to discard of suspender liquors	1.5%	0.7%
(2) Loss due to discard of spent tan	1.5	1.5
(3) Loss due to fermentation and bloom formation	4.0	23.0
(4) Loss due to sludge formation (as distinct from bloom formation)	10.0	1.8
(5) Loss due to floor losses, overflows and accidents	2.0	2.0
(6) Loss due to discard of wash liquors	1.0	1.0
	20.0%	30.0%

Any attempt to prevent fermentation by sterilization is dangerous. Sludge may be reduced by excessive sulfiting. American sole leather tanners avoid much of the trouble this way. However the leather produced, although good according to American standards, would not suit many British customers. Addition of sulfite cellulose or some syntans is a helpful treatment of some deposit-liable liquors. This leads naturally to this question, would it be possible to synthesize a satisfactory product for this express purpose?

The Effect of Drying on the Nature of the Acid Groups in Chrome-tanned Collagen. By J. C. de Wijs. *J. Soc. Leather Trades' Chemists*, **39**, 381 (1955). A 33% basic chromium sulfate tanning solution was prepared by dissolving hexaquochromium sulfate and adjusting the basicity with NaOH. The solution was boiled ten minutes, cooled, diluted, and aged for at least a week. Chrome liquors were also prepared using sodium formate and phthalate. Hide powder was tanned to contain 4.5-5.0% Cr_2O_3. The tanned powder was washed, suspended in distilled water, and neutralized with NaOH to a pH of 5.3-5.7. In other experiments the tanned powder was not neutralized. The chrome powders had moisture contents of approximately 75%. The ionic acid content was determined (by Gustavson's pyridine method) in the chrome-tanned powders before and after drying to various moisture contents. Drying of neutralized chrome powder did not alter the amount of ionic-bound acid; whereas, in the nonneutralized powder the acid decreased progressively as the drying continued. It is concluded that (1) in nonneutralized leather ionic acid groups exterior to the chromium complex enter into the complex when the leather is dried, and (2) in neutralized leather the ionic acid groups of the chromium complex are completely removed, leaving a neutral (or slightly anionic) complex. J.M.C.

The Diffusion of Water Vapor in Leather. By R. G. Mitton. *J. Soc. Leather Trades' Chemists*, **39**, 385 (1955). Transmission of water vapor through leather may in principle occur in two distinct ways: by vapor diffusion in the air within capillary spaces in the leather, and by diffusion within the solid fibers themselves. Since the relative disadvantages of different waterproofing treatments are dependent on the mechanism of vapor transfer (i.e., shoe leather is desired to be impermeable to liquid yet permeable to vapor), the relative importance of the two mechanisms of vapor transfer should be known. Diffusion of water vapor through leather was studied by maintaining at its faces two different concentrations of vapor. After a steady state was attained, i.e., rate of transfer of moisture into leather same as that escaping from the opposite side, weight loss was plotted aganist time and rate of loss of weight, corrected for thickness; and cross-sectional area of the

leather plug, was plotted against porosity. By either plot, diffusion within the fibers was found to be insignificant. Measurements were also made in a "nonsteady state" by maintaining the vapor concentration at one face constant, preventing moisture from entering or leaving at the other, and then following the moisture content of the sample as a function of time. Adsorption was measured in leather plugs of different lengths; with plugs of various densities of packing; at temperatures of 21 and 50°C; and in atmospheres of different humidities. None of the experiments gave an indication that diffusion through the solid fiber accounts for any appreciable transfer of moisture. The most sensitive experiment indicated that in air-dry sole leather at least 97 per cent and in ordinary upper leather at least 99% of the moisture transferred diffuses through the capillary spaces. Diffusion through the fibers may become more important at high humidities. In nonsteady-state diffusion the capacity of the fibers to absorb moisture greatly affects transport of moisture even though diffusion occurs almost entirely in the gas. Taking into account both moisture absorbed and that transmitted, leather can remove from the interior of a shoe much more perspiration than could a material of similar porosity which had no absorptive capacity.
J.M.C.

Our Present Knowledge of Collagen. (Max Bergman Memorial Lecture at the 4th Biennial Conference of the International Union of Leather Chemists Societies, Aug. 1, 1955 in Stockholm.) By W. Grassmann. *Das Leder*, **6**, 241 (1955). A review of (1) X-ray interference and the theory of a spiral structure of the fibers, (2) discovery and investigation of small angle interference, (3) discovery and investigation of electron microscopic striations (4) investigations of soluble collagen fibers and (5) chemical and enzymatic decomposition and the amino acid sequence in collagen. 103 references. I.D.C.

Beamhouse Problems in the Production of Vegetable-tanned, Split Leather. By H. Gnamm. *Das Leder*, **6**, 265 (1955). General introduction to the "Discussion Day" of the VGCT in Darmstadt 9-16-55, and to the panel discussion on split leather. The soaks and limes must be adjusted for the principal type of leather being made. Softer leather for upholstery can be made by reliming. Conditions for tight grain and for faultless white hide are antagonistic, so a careful adjustment to a narrow intermediate zone is necessary.
I.D.C.

Beamhouse Problems in the Production of Sole Leather. By K. Löchner. *Das Leder*, **6**, 275 (1955). Introduction to a panel discussion. Liming is less critical for sole than upper leather. Only well limed hides will make first class leather but poorly limed hides may still make good sole leather. Limes sharpened with sulfide (amines are not used in Germany) are used if the hair is to be saved, but this requires more handling and hair is cheap, so it is often "burned" off with 4 per cent lime, 2 per cent sodium sulfide in a 300 per cent float. Soaking and unhairing should be done in the same vessel to save labor. U.S.A. hides are high in fat so it is necessary to use limes at a higher temperature than normal and to add additional sulfide.
I.D.C.

Identification of a New Compound (Lanthionine sulfoxide?) in Products from Animal Skin. By H. Zahn and D. Wegerle. *Das Leder*, **6**, 278 (1955). In 1953 a paper chromatogram of a gelatin hydrolysate showed a weak ninhydrin-positive spot, which had a mobility (R_f) with respect to lysine (R_{Lys}) of 0.15. Later, after synthesis of lanthionin sulfoxide a comparison of chromatograms indicated the unknown was probably lanthionin sulfoxide. Meanwhile the ninhydrin-positive compound with R_{Lys}=0.16-.20 had been found in decomposition products isolated from a lime liquor and also in the hydrolysate from long-limed glue stock. It was assumed that the sulfoxide was formed by the action of alkali

and oxidation on the cystine from hair and epidermis. Isolation of the sulfoxide will be necessary to confirm this identification. In other products spots with the following R_{Lys} values were found: I. lime liquor, weak-medium, 0.19; II. lime liquor, weak, 0.18-.19; III. keratose, weak, 0.16-.17; IV. peroxide-keratose, medium, 0.20; V. hair keratose, medium-strong, 0.20; VI. paste, very weak, 0.16. These materials were prepared as follows: I. 100 ml. of a spent enzyme lime liquor containing sodium hydroxide and sulfhydrate from calfskins was evaporated at 90° and the residue dried over sufuric acid and potassium hydroxide. II. A grain-free split was limed for 50 days at room temperature, then 250 ml. of the liquor was dialyzed, adjusted to pH 5 with normal hydrochloric acid and freeze dried. III. Normal limes from cattle or horse hides or calfskins were neutralized, the precipitate was extracted with sodium hydroxide solution, then keratose was precipitated at the isoelectric point, reprecipitated and dried at 35°. IV. A calfskin lime liquor was treated with acid, the precipitated mixture of protein, sulfur and polysulfides was dissolved in alkali, treated with barium peroxide, then precipitated with acid. V. Calf hair was relimed in a solution containing sodium hydroxide and sulfhydrate, then the liquor was treated as for III. VI. Glue stock was limed for 35 days, washed with water and dilute acid, brought to pH 3, then shredded to a paste containing 90 percent water. I.D.C.

Beamhouse Problems in the Production of Boxcalf. By A. Zissel. *Das Leder*, **6**, 289 (1955). Introduction to a round table discussion. In liming for boxcalf leather, the hair should be well loosened, but preserved. This can be obtained by painting or multiple liming. Short, sharp limes open up the skin with sufficient preservation of hide substance. A firm, smooth, fine grain is best obtained by painting. Old liquors and higher temperatures produce a fine grain, but can cause loose grain. Drawn grain is caused by changes in alkalinity and temperature during liming, but can be reduced by mild liming conditions. In general, higher temperatures during liming and green fleshing are favorable. I.D.C.

Beamhouse Problems in the Production of Chrome Side Leather. By F. Tacheci. *Das Leder*, **6**, 295 (1955). Introduction to a round table discussion. In a process, soaking, liming, mechanical working, and bating should be balanced. Salt should be removed as completely as possible at the beginning of soaking. European tanners prefer a warm, short liming to the American long, mild liming. I.D.C.

PATENTS

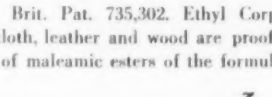
Lignin-Tanned Leather and Process for Making Same. U. S. Pat. 2,729,534. Hans Ehrensperger, Mackayville, Quebec, Canada. Appl. Sept. 7, 1955.

1. A process of producing leather comprising soaking leather stock in an aqueous solution of lignin-containing material selected from the group consisting of sulphonic acid-lignin and alkali lignin, said aqueous solution having a pH above about 3.5 in the case of sulphonic

acid-lignin, said aqueous solution having a pH above about 6.5 in the case of alkali lignin when said alkali lignin is kraft lignin, and said aqueous solution having a pH above about 8.5 in the case of alkali lignin when said alkali lignin is not kraft lignin, and a water-soluble condensation product of a water-soluble aldehyde with a member of the group consisting of monomeric double acid amides and amino triazines, then acidifying the solution without heating to precipitate the active substance in situ as a water-insoluble precipitate, said acidification being to a pH below about 2.5 when said lignin material is a sulphonic acid lignin and to a pH below about 5 when said lignin material is an alkali lignin.

Tanning with Tetrakis-(Hydroxymethyl) Phosphonium Chloride. U. S. Pat. 2,732,278. Edward M. Filachione, Philadelphia, Pa., assignor to the United States of America as represented by the Secretary of Agriculture. Appl. Mar. 22, 1955. (Granted under Title 35, U. S. Code (1952), sec. 266). 1. A process for tanning hides comprising treating the hides with tetrakis-(hydroxymethyl) phosphonium chloride at a pH of about 2 to 12.

Proofing Against Fungi. Brit. Pat. 735,302. Ethyl Corporation. Appl. May 1, 1952. Permeable materials such as cloth, leather and wood are proofed against fungi by treatment with organic solvent solutions of maleamic esters of the formula



where R is alkyl, aralkyl, alkenyl, or a halogen-, sulphur-, or nitrogen-substituted derivative thereof, X and Y are hydrogen or halogen, Z is an organic group or a negative group non-reactive with an aliphatic alcohol and n is 1—5 subject to the following limitations: (1) Z is organic when both X and Y are hydrogen; (2) when X and Y are different the position of the halogen atom is the same as that in the maleamic acid obtained by the reaction of a halomaleic anhydride with an aromatic amine; (3) N-(*p*-tolyl)-bromomaleamic methyl ester is excluded. Various hydrocarbons, ketones, chlorine compounds, esters, alcohols and glycol monoethers are specified as suitable organic solvents.

Depilatory Paints. Brit. Pat. 735,783. Zalcman H. Appl. Feb. 17, 1953. A depilatory paint of the type used for dewooling sheepskins and the dehairing of other skins for application to the flesh side of skins comprises an aqueous composition free from monovalent cations made up from a source of hydroxyl ions selected from the oxides and hydroxides of calcium, strontium and barium, a source of hydrosulphide ions selected from the sulphides and hydrosulphides of calcium, magnesium, strontium, aluminium and barium, and a reserve source of hydroxyl ions, the hydrosulphide ion content being within the range equivalent to 2.0 to 23.5 per cent calcium hpdrosulphide by weight of the composition, and the hydroxyl ion concentration being such that the *p*H is more than 12.07 and not more than 12.3. A water-soluble calcium salt may be added if necessary to depress the hydroxyl ion concentration. The composition may contain an inert filler such as kaolin, kieselguhr, talc or cereal flour. The hydroxyl ion concentration can be reduced by the addition of calcium chloride for example, or a soluble salt of a polyvalent metal which will react with lime to form a soluble calcium salt and precipitate the polyvalent metal hydroxide. Aqueous compositions exemplified contain (i) calcium hydrosulphide and lime with barium or strontium hydroxides; (ii) calcium hydrosulphide and lime with barium or strontium sulphide; (iii) calcium hydrosulphide, barium or strontium hydroxide and a water-soluble calcium salt; (iv) barium or strontium sulphide and lime with a water-soluble calcium salt; (v) barium

or strontium hydrosulphide and lime with a water-soluble calcium salt; (vi) barium or strontium hydrosulphide and barium or strontium hydroxide with a water-soluble calcium salt. Compositions (i) and (ii) are preferred. The paint compositions are suitable for depilating sheepskins at 70° to 80° F. in 24 hours, or longer in the case of hides. The hides or skins are preferably soaked and fleshed before depilation. Specifications 680,784 and 706,746 are referred to.

Waterproofing Leather. Brit. Pat. 736,378. Midland Silicones, Ltd., [formerly Dow Corning, Ltd.]. Appl. June 18, 1953. Leather is waterproofed by impregnation with a composition comprising (1) 15 to 50 per cent (by weight) of $Ti(OR)_4$ or an aliphatic hydrocarbon-soluble partial hydrolysate thereof in which R is an aliphatic hydrocarbon radical having less than 13 carbon atoms or a hydroxylated aliphatic hydrocarbon radical having less than 13 carbon atoms and containing less than 4 hydroxy radicals; (2) 5 to 70 per cent of a copolymer composed of trimethylsiloxane and SiO_2 units, the units being in such proportion that the CH_3 : Si ratio is from 1.0 : 1 to 2.5 : 1, and (3) 15 to 80 per cent of a polysiloxane of the formula

$$R^1_nSiO_{\frac{4-n}{2}}$$

where R^1 is an alkyl or alkenyl radical having less than 4 carbon atoms, or a monocyclic aryl radical, and n has an average value of 2 to 2.9 (*see* Group IV (a)). The compositions may be used in the form of solvent-less pastes which may contain polishing agents, or in solution in non-aromatic solvents such as Stoddard solvent or naphtha mineral spirits. The compositions may be applied by brushing or dipping leather sheet or articles such as shoes or belts. Any leather, tanned, tawed or otherwise cured, finished or unfinished, or leather from horse hide, pig-skin and other animals, may be treated. The leather used in the examples is "Tomahawk Kip," a commercial emulsion top vegetable tanned cowhide.

Waterproofing Leather. Brit. Pat. 736,584. Armour & Co. Appl. Feb. 9, 1951. Leather is permanently waterproofed by reaction with an oxidizing agent capable of hydroxylating the free amino groups of the protein and then with a fatty acid halide having from 1 to 22 carbon atoms in its hydrocarbon radical. Suitable oxidizing agents mentioned are hydrogen peroxide, peracetic acid and permonosulphuric acid and fatty acid halides mentioned include lauryl, palmityl, oleyl, tallow acid and stearyl chlorides.

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INDEX TO ADVERTISERS

ADOLF SCHUBERT XXXV
AMERICAN CYANAMID COMPANY, Dyestuff Department XIII
AMERICAN EXTRACT CO. ..... Outside Back Cover
ANTARA CHEMICALS III
APEX CHEMICAL CO., INC. XXXII
ARKANSAS COMPANY, INC. VII
ARTHUR C. TRASK CO. XXXI
ARTHUR THOMAS CO. XXII
ATLAS REFINERY XVI
BARIUM REDUCTION CORPORATION XXV
BARKEY IMPORTING CO., INC. XIV
BONA ALLEN, INC. XXXIV
BUCKMAN LABORATORIES, INC. XII
CHEMTAN COMPANY XXI
CLINTON FOODS XXXIV
EISENDRATH TANNING CO. XXVII
EUROPEAN CHESTNUT MFG'S ASSO. XVII
FRANK F. MARSHALL XXXI
GARDEN STATE TANNING INC. XXXI
GEIGY COMPANY, INC. XI
HOOKER ELECTROCHEMICAL CO. XIX
HOWES LEATHER CO., INC. XXXIV
INTERNATIONAL PRODUCTS CORP. IX
KEPEC CHEMICAL CORPORATION XXXI
LEATHER AND SHOES X
LEATHER MANUFACTURER, THE XXIX
L. H. LINCOLN & SON, INC. I
MARDEN WILD CORPORATION XXVI
MUTUAL CHEMICAL DIVISION, ALLIED CHEMICAL & DYE CORP. ..... Inside Back Cover
NEWARK LEATHER FINISH CO. XXXIII
NOPCO CHEMICAL CO. XXIII
OHIO LEATHER CO., THE XXVII
OLSON SALES AGENCY, THE ..... Inside Front Cover
PACIFIC COAST BORAX CO. XXVIII
PRESTO COLOR CO. XXVIII
REILLY-WHITEMAN-WALTON CO. XXIX
RIVER PLATE CORPORATION, THE V
ROBESON PROCESS CO. II
ROHM & HAAS IV
SALEM OIL & GREASE CO. XXX
SAXE CUTCH CORPORATION XXVI
SHOE & LEATHER REPORTER XXX
SOLVAY PROCESS DIVISION, ALLIED CHEMICAL & DYE CORP. VI
SOUTH AFRICAN WATTLE EXTRACT MFRS. ASSO. XV
SUN OIL COMPANY XXVI
TANEXCO, INC. XXXVIII
TANIMEX CORPORATION XVIII
TANNER'S COUNCIL RESEARCH LABORATORY ..... XXXI—XXXV—XXXVI
UNION STARCH & REFINING CO. XXXII
UNITED STATES TESTING COMPANY XXXIII
WALLERSTEIN COMPANY, INC. XXIV
WARNER CO. XXVII
WHITTEMORE-WRIGHT CO. XXVIII
WOLF, JACQUES & CO. VIII
YOUNG CO., J. S. XX

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549 WEST WASHINGTON BLVD., CHICAGO 6, ILLINOIS

SOLE AGENTS
U. S. A.

Mutual's name for their basic chromic sulfate used in one-bath chrome tanning. Available in two grades with approximate analyses as shown below.

	Koreon M	Koreon X
Cr_2O_3	24%	25%
Basicity (Schorlemmer)	33%	52%

Koreon is packed in multi-wall paper bags containing 75 pounds, and in fibre drums containing about 325 pounds.

SODIUM BICHROMATE POTASSIUM BICHROMATE

MUTUAL CHEMICAL DIVISION

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